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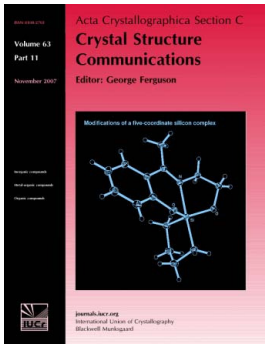

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Eight salt forms of sulfadiazine

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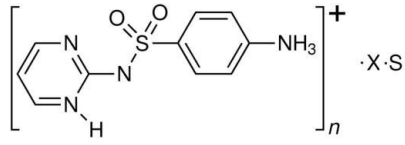
Proton transfer to the sulfa drug sulfadiazine [systematic name: 4-amino-*N*-(pyrimidin-2-yl)benzenesulfonamide] gave eight salt forms. These are the monohydrate and methanol hemisolvate forms of the chloride (2-{[(4-azaniumylphenyl)sulfonyl]azanidyl}pyrimidin-1-ium chloride monohydrate, $C_{10}H_{11}N_4O_2S^+\cdot Cl^-\cdot H_2O$, (I), and 2-{[(4-azaniumylphenyl)-sulfonyl]azanidyl}pyrimidin-1-ium chloride methanol hemisolvate, $C_{10}H_{11}N_4O_2S^+\cdot Cl^-\cdot 0.5CH_3OH$, (II)); a bromide monohydrate (2-{[(4-azaniumylphenyl)sulfonyl]azanidyl}pyrimidin-1-ium bromide monohydrate, $C_{10}H_{11}N_4O_2S^+\cdot Br^-\cdot H_2O$, (III)), which has a disordered water channel; a species containing the unusual tetraiodide dianion [bis(2-{[(4-azaniumylphenyl)-sulfonyl]azanidyl}pyrimidin-1-ium) tetraiodide, $2C_{10}H_{11}N_4O_2S^+\cdot I_4^{2-}$, (IV)], where the $[I_4]^{2-}$ ion is located at a crystallographic inversion centre; a tetrafluoroborate monohydrate (2-{[(4-azaniumylphenyl)sulfonyl]azanidyl}pyrimidin-1-ium tetrafluoroborate monohydrate, $C_{10}H_{11}N_4O_2S^+\cdot BF_4^-\cdot H_2O$, (V)); a nitrate (2-{[(4-azaniumylphenyl)sulfonyl]azanidyl}pyrimidin-1-ium nitrate, $C_{10}H_{11}N_4O_2S^+\cdot NO_3^-$, (VI)); an ethanesulfonate {4-[(pyrimidin-2-yl)sulfamoyl]anilinium ethanesulfonate, $C_{10}H_{11}N_4O_2S^+\cdot C_2H_5SO_3^-$, (VII)}; and a dihydrate of the 4-hydroxybenzenesulfonate {4-[(pyrimidin-2-yl)sulfamoyl]anilinium 4-hydroxybenzenesulfonate dihydrate, $C_{10}H_{11}N_4O_2S^+\cdot HOC_6H_4SO_3^-\cdot 2H_2O$, (VIII)}. All these structures feature alternate layers of cations and of anions where any solvent is associated with the anion layers. The two sulfonate salts are protonated at the aniline N atom and the amide N atom of sulfadiazine, a tautomeric form of the sulfadiazine cation that has not been crystallographically described before. All the other salt forms are instead protonated at the aniline group and on one N atom of the pyrimidine ring. Whilst all eight species are based upon hydrogen-bonded centrosymetric dimers with graph set $R_2^2(8)$, the two sulfonate structures also differ in that these dimers do not link into one-dimensional chains of cations through NH_3-to-SO_2 hydrogen-bonding interactions, whilst the other six species do. The chloride methanol hemisolvate and the tetraiodide are isostructural and a packing analysis of the cation positions shows that the chloride monohydrate structure is also closely related to these.

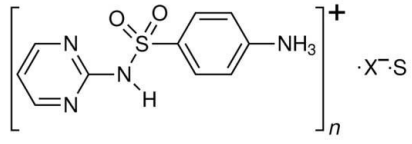
Keywords: crystal structure; sulfa drugs; sulfadiazine; salt forms; tautomerism; pharmaceutical compounds; tetraiodide; active pharmaceutical ingredients (APIs).

1. Introduction

Sulfadiazine [systematic name: 4-amino-*N*-(pyrimidin-2-yl)-benzenesulfonamide] is a sulfonamide antibiotic which is often used in the form of an Ag^I complex within a topical cream (Fisher *et al.*, 2003). Although crystal structures of both sulfadiazine and its Ag^I complex have been known for some time (Kokila *et al.*, 1995; Cook & Turner, 1975), and despite the general interest in modifying physicochemical properties of Active Pharmaceutical Ingredients (APIs) by salt formation (Stahl & Wermuth, 2008), it is only recently that Englert and co-workers described the first structures of salt forms containing protonated sulfadiazine cations (Pan *et al.*, 2013). That study on two solvated forms of sulfadiazine hydrochloride used charge-density measurements to probe the



(I) X = Cl^-, *n* = 1, S = H_2O
(II) X = Cl^-, *n* = 1, S = 0.5 MeOH
(III) X = Br^-, *n* = 1, S = H_2O
(IV) X = I_4^{2-}, *n* = 2
(V) X = BF_4^-, *n* = 1, S = H_2O
(VI) X = NO_3^-, *n* = 1



(VII) X = $EtSO_3$, *n* = 1
(VIII) X = $HOC_6H_4SO_3$, *n* = 1, S = $2H_2O$

unusual protonation behaviour of sulfadiazine – which was shown to form cations by protonating two N atoms of the aniline group and the pyrimidine ring whilst leaving the amide N atom unprotonated and formally carrying a negative charge. This is contrary to what may be expected based on pK_a values and to what has been observed for cations of the related sulfonamide drug sulfamethazine (Smith & Wermuth, 2013*a*,*b*; Lu *et al.*, 2008), where the weakly basic aniline group remains neutral as an NH_2 species. In order to further examine this feature, we extend the number of sulfadiazine salt structues known by presenting eight new structures, *viz.* compounds (I)–(VIII) (see Scheme).

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Table 1
Experimental details.

For all refinements, H atoms were treated by a mixture of independent and constrained refinement.

	(I)	(II)	(III)	(IV)
Crystal data				
Chemical formula	$C_{10}H_{11}N_4O_2S^+\cdot Cl^-\cdot H_2O$	$C_{10}H_{11}N_4O_2S^+\cdot Cl^-\cdot 0.5CH_4O$	$C_{10}H_{11}N_4O_2S^+\cdot Br^-\cdot H_2O$	$2C_{10}H_{11}N_4O_2S^+\cdot I_4^{2-}$
M_r	304.75	302.76	349.21	1010.18
Crystal system, space group	Triclinic, $P\overline{1}$	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/n$	Triclinic, $P\overline{1}$
Temperature (K)	100	100	123	123
a, b, c (Å)	5.4118 (4), 11.5632 (8), 11.7430 (8)	5.6468 (2), 11.2749 (4), 11.8363 (8)	11.9420 (3), 5.6295 (2), 21.0659 (6)	5.7792 (4), 12.0002 (7), 12.5214 (9)
α, β, γ (°)	109.462 (7), 94.399 (6), 102.509 (7)	64.254 (5), 79.361 (6), 77.687 (6)	90, 98.380 (3), 90	61.964 (7), 87.745 (5), 76.426 (5)
V (Å^3)	667.51 (8)	659.53 (6)	1401.09 (7)	742.51 (9)
Z	2	2	4	1
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.45	0.45	3.09	4.38
Crystal size (mm)	0.06 × 0.02 × 0.01	0.09 × 0.06 × 0.05	0.30 × 0.22 × 0.04	0.25 × 0.15 × 0.02
Data collection				
Diffractometer	Rigaku Saturn724+ (2×2 bin mode) diffractometer	Rigaku Saturn724+ (2×2 bin mode) diffractometer	Oxford Diffraction Xcalibur E diffractometer	Oxford Diffraction Xcalibur E diffractometer
Absorption correction	Multi-scan (*CrystalClear-SM Expert*; Rigaku, 2013)	Multi-scan (*CrystalClear-SM Expert*; Rigaku, 2013)	Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)	Analytical [*CrysAlis PRO* (Oxford Diffraction, 2010), based on expressions derived by Clark & Reid (1995)]
T_{min}, T_{max}	0.897, 1.000	0.819, 1.000	0.616, 1.000	0.523, 0.905
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8329, 3000, 2752	11731, 3028, 2804	8186, 3353, 2868	7055, 3595, 2996
R_{int}	0.025	0.029	0.024	0.033
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.649	0.649	0.661	0.691
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.079, 1.05	0.031, 0.082, 1.04	0.029, 0.072, 1.06	0.032, 0.062, 1.04
No. of reflections	3000	3028	3353	3595
No. of parameters	197	201	203	184
No. of restraints	5	1	5	0
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.51, −0.38	0.40, −0.43	0.46, −0.52	0.66, −0.93

	(V)	(VI)	(VII)	(VIII)
Crystal data				
Chemical formula	$C_{10}H_{11}N_4O_2S^+\cdot BF_4^-\cdot H_2O$	$C_{10}H_{11}N_4O_2S^+\cdot NO_3^-$	$C_{10}H_{11}N_4O_2S^+\cdot C_2H_5O_3S^-$	$C_{10}H_{11}N_4O_2S^+\cdot C_6H_5O_4S^-\cdot 2H_2O$
M_r	356.11	313.30	360.41	460.48
Crystal system, space group	Monoclinic, $P2_1/n$	Triclinic, $P\overline{1}$	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/n$
Temperature (K)	123	123	123	123
a, b, c (Å)	5.8539 (1), 11.3629 (3), 21.2080 (6)	5.4705 (4), 10.1235 (7), 11.9956 (8)	5.5269 (4), 34.979 (3), 8.4395 (6)	18.5872 (5), 5.9733 (2), 18.6850 (5)
α, β, γ (°)	90, 90.982 (2), 90	86.708 (6), 85.489 (6), 74.394 (7)	90, 93.102 (7), 90	90, 104.858 (3), 90
V (Å^3)	1410.49 (6)	637.40 (8)	1629.2 (2)	2005.17 (10)
Z	4	2	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.30	0.29	0.36	0.32
Crystal size (mm)	0.20 × 0.12 × 0.05	0.26 × 0.15 × 0.06	0.4 × 0.03 × 0.02	0.20 × 0.12 × 0.05
Data collection				
Diffractometer	Oxford Diffraction Xcalibur E diffractometer	Oxford Diffraction Xcalibur E diffractometer	Oxford Diffraction Xcalibur E diffractometer	Oxford Diffraction Xcalibur E diffractometer
Absorption correction	Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)	Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)	Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)	Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min}, T_{max}	0.874, 1.000	0.897, 1.000	0.908, 1.000	0.968, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8710, 3549, 2955	7390, 2977, 2429	19991, 3515, 2212	9653, 4695, 3638
R_{int}	0.024	0.026	0.104	0.029
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.682	0.661	0.639	0.661
Refinement				
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.089, 1.03	0.045, 0.110, 1.04	0.055, 0.112, 1.01	0.048, 0.113, 1.03
No. of reflections	3549	2977	3515	4695
No. of parameters	232	206	222	307
No. of restraints	3	0	3	6
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.35, −0.46	0.78, −0.38	0.35, −0.41	0.80, −0.44

Computer programs: *CrystalClear-SM Expert* (Rigaku, 2013), *CrysAlis PRO* (Oxford Diffraction, 2010), *SIR92* (Altomare *et al.*, 1994), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008) and *X-SEED* (Barbour, 2001).

2. Experimental

2.1. Synthesis and crystallization

Following the method of Buist *et al.* (2013), chloride methanol hemisolvate (II) was prepared by adding acetyl chloride (1 ml) to a methanol solution (4 ml) of sulfadiazine (0.226 g). Crystals were grown directly from the reaction mixture by slow evaporation of the solvent. All other compounds were obtained by adding the appropriate acid to aqueous solutions of sulfadiazine in water, filtering to obtain clear solutions, and then allowing slow evaporation of the solvent to promote crystal growth. Solid-state IR spectra were measured with an A_2 Technologies ATR instrument and are included in the *Supporting information.*

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. For all eight title structures, H atoms bound to C atoms were placed in the expected geometric positions and treated in riding modes, with C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C), except for methyl H atoms, for which C—H = 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C). With the exceptions noted below, H atoms bound to O or N atoms were placed as found and refined isotropically. For all hydrates, the O—H and H···H distances of the water molecules were restrained to 0.88 (1) and 1.33 (2) Å, respectively. Similarly, the O—H distance of the methanol solvent molecule in (II) was restrained to 0.88 (1) Å and the azanium N—H distances in (VII) were restrained to 0.91 (1) Å. For (I), (II) and (III), the solvent H atoms were also given displacement parameters such that U_{iso}(H) = 1.2U_{eq}(O). For (I), atoms H2*W* and H3*W* have site-occupancy factors of 0.5. For tetraiodide (IV), it was found neccessary to set the U_{iso}(H) values at 1.2U_{eq}(N) for the H atoms bound to N atoms. For (III), the water molecule was found to be disordered over several sites that formed a channel. In the final structure, after numerous trial calculations, this was modelled as four O-atom sites. These were constrained to have equal displacement parameters and their individual site-occupancy parameters were allowed to refine independently but were restrained to sum to unity. This gave final values of 0.453 (10), 0.248 (10), 0.161 (6) and 0.139 (4) for O1*W*, O2*W*, O3*W* and O4*W*, respectively. For these disordered sites, H atoms could only be placed on the major component, *i.e.* O1*W*, with U_{iso}(H) = 1.5U_{eq}(O). For the amide H atom in (III), U_{iso}(H) values were set at 1.2U_{eq}(N) and for the anilinium groups in (III) and (VII) they were set at 1.5U_{eq}(N).

3. Results and discussion

Two chloride-containing salt phases were prepared, *i.e.* a chloride monohydrate, (I) (Fig. 1), obtained from aqueous solution and a methanol hemisolvate, (II) (Fig. 2), obtained by reacting sulfadiazine with acetyl chloride in methanol [see Buist *et al.* (2014) for another example of the use of these reagents for obtaining differently solvated forms of APIs]. Methanol hemisolvate (II) is isostructural with the ethanol and ethylene glycol solvates described previously by Pan *et al.* (2013). All three isostructural forms feature a solvent molecule at a crystallographic inversion centre. For the methanol hemisolvate, (II), and ethanol species this results in a disordered solvent site with half-occupancy atoms. Chloride monohydrate (I) also has a very similar structure, as can be shown by constructing 20-molecule overlays in *Mercury* (Macrae *et al.*, 2008; Allen, 2002). These confirm that all four chloride forms have 20 out of 20 cation positions in common (when software option 'ignore smallest molecular components' is selected and 30% geometric tolerances are allowed). For (I), the smaller solvent size means that two water molecules can be accommodated at the solvent site with no O-atom disorder. The H atoms of the water molecule are, however, disordered, with H2*W* and H3*W* being alternative sites both with site-occupancy factors of 0.5. These disordered H atoms take part in water-to-water hydrogen-bond contacts which give polymeric chains of connected water molecules that extend parallel to the crystallographic *a* direction. The remaining well-ordered H atom, H1*W*, forms a hydrogen bond to the chloride ion. The three organic solvates cannot form

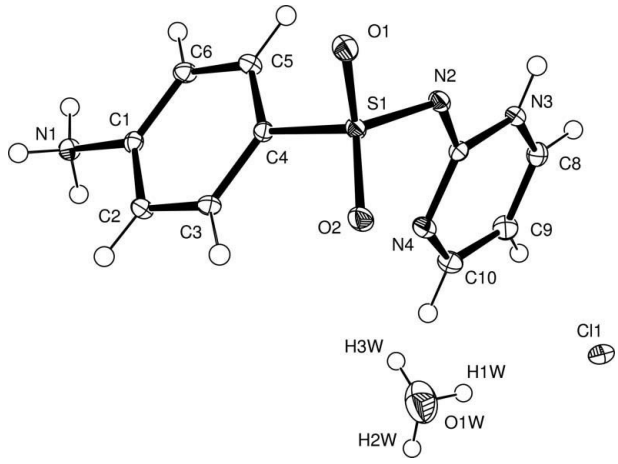

Figure 1
The molecular structure of (I), with non-H atoms shown as 50% probability displacement ellipsoids. The H2*W* and H3*W* sites are disordered and both have site-occupancy factors of 0.5.

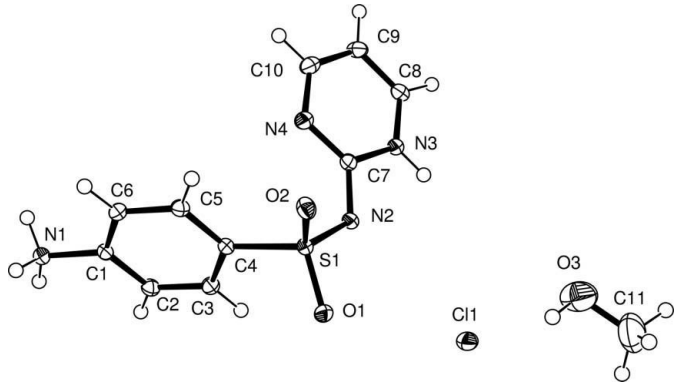

Figure 2
The molecular structure of (II), with non-H atoms shown as 50% probability displacement ellipsoids. All atoms of the methanol solvent molecule have site-occupancy factors of 0.5.

Table 2
Hydrogen-bond geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···Cl1^{i}	0.91 (2)	2.27 (2)	3.1484 (13)	161.8 (19)
N1—H2N···Cl1^{ii}	0.91 (2)	2.27 (2)	3.1795 (13)	176.0 (18)
N1—H3N···O1^{iii}	0.91 (2)	1.99 (2)	2.8243 (16)	150.2 (19)
N3—H4N···N2^{iv}	0.90 (2)	1.98 (2)	2.8763 (17)	175.9 (19)
N3—H4N···O1^{iv}	0.90 (2)	2.54 (2)	3.0962 (15)	121.1 (16)
O1*W*—H1*W*···Cl1	0.88 (1)	2.62 (1)	3.4607 (17)	160 (3)
O1*W*—H2*W*···O1*W*^{v}	0.88 (1)	2.18 (2)	2.995 (4)	153 (4)
O1*W*—H3*W*···O1*W*^{i}	0.88 (1)	2.21 (2)	3.030 (4)	155 (5)

Symmetry codes: (i) $-x+2, -y+2, -z+1$; (ii) $-x+1, -y+2, -z+1$; (iii) $-x, -y+1, -z$; (iv) $-x, -y+1, -z+1$; (v) $-x+3, -y+2, -z+1$.

Table 3
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···Cl1^{i}	0.91 (2)	2.30 (2)	3.2088 (14)	174.1 (18)
N1—H2N···Cl1^{ii}	0.86 (2)	2.70 (2)	3.2151 (13)	119.8 (18)
N1—H2N···O1^{ii}	0.86 (2)	2.13 (2)	2.8701 (18)	144 (2)
N1—H3N···Cl1^{iii}	0.92 (3)	2.25 (3)	3.1417 (14)	163 (2)
N3—H4N···N2^{iv}	0.89 (2)	2.01 (2)	2.8957 (18)	175.4 (19)
N3—H4N···O1^{iv}	0.89 (2)	2.59 (2)	3.1218 (16)	119.6 (16)
O3—H1H···Cl1	0.89 (1)	2.31 (2)	3.170 (3)	163 (6)

Symmetry codes: (i) $x, y+1, z$; (ii) $-x+1, -y+2, -z$; (iii) $x-1, y+1, z$; (iv) $-x+1, -y+1, -z+1$.

Table 4
Hydrogen-bond geometry (Å, °) for (III).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···Br1	0.91 (3)	2.44 (3)	3.308 (2)	159 (2)
N1—H2N···O2^{i}	0.88 (3)	2.01 (3)	2.810 (3)	150 (3)
N1—H2N···Br1^{ii}	0.88 (3)	2.95 (3)	3.4022 (18)	114 (2)
N1—H3N···Br1^{iii}	0.85 (3)	2.46 (3)	3.317 (2)	178 (2)
N3—H4N···N2^{iv}	0.87 (1)	2.02 (1)	2.887 (2)	179 (2)
N3—H4N···O2^{iv}	0.87 (1)	2.57 (2)	3.080 (2)	119 (2)

Symmetry codes: (i) $-x+1, -y+2, -z+2$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x, y+1, z$; (iv) $-x+2, -y+2, -z+2$.

Table 5
Hydrogen-bond geometry (Å, °) for (IV).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···I1^{i}	0.85 (4)	2.64 (4)	3.482 (4)	172 (4)
N1—H2N···O2^{ii}	0.84 (4)	2.00 (4)	2.798 (4)	158 (4)
N1—H3N···I1	0.96 (4)	2.59 (4)	3.524 (3)	165 (3)
N4—H4N···N2^{iii}	0.91 (4)	2.00 (4)	2.906 (5)	174 (4)

Symmetry codes: (i) $x+1, y, z$; (ii) $-x, -y+2, -z+1$; (iii) $-x, -y+2, -z+2$.

Table 6
Hydrogen-bond geometry (Å, °) for (V).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···F3^{i}	0.93 (2)	1.92 (2)	2.7912 (17)	156.0 (19)
N1—H2N···O2^{ii}	0.88 (2)	2.05 (2)	2.8196 (19)	144.7 (18)
N1—H3N···O1*W*	0.92 (2)	1.87 (3)	2.7762 (19)	166 (2)
N3—H4N···N2^{iii}	0.89 (2)	2.02 (2)	2.9092 (17)	176.8 (19)
N3—H4N···O2^{iii}	0.89 (2)	2.58 (2)	3.1307 (17)	121.2 (15)
O1*W*—H1*W*···F4^{iv}	0.87 (1)	2.03 (1)	2.8790 (15)	165 (2)
O1*W*—H2*W*···F1	0.88 (1)	1.94 (1)	2.7863 (17)	163 (2)

Symmetry codes: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x, -y+1, -z+2$; (iii) $-x, -y, -z+2$; (iv) $x-1, y, z$.

Table 7
Hydrogen-bond geometry (Å, °) for (VI).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O5^{i}	0.96 (3)	1.85 (3)	2.803 (3)	171 (3)
N1—H1N···O4^{i}	0.96 (3)	2.42 (3)	2.968 (3)	116 (2)
N1—H2N···O3	0.85 (3)	2.31 (3)	2.912 (3)	128 (3)
N1—H2N···O5	0.85 (3)	2.32 (3)	3.021 (3)	140 (3)
N1—H3N···O1^{ii}	0.95 (3)	1.85 (3)	2.784 (2)	166 (3)
N3—H4N···N2^{iii}	0.85 (3)	2.06 (3)	2.911 (3)	176 (2)
N3—H4N···O1^{iii}	0.85 (3)	2.55 (3)	3.085 (2)	122 (2)

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y, -z+1$; (iii) $-x+1, -y, -z+2$.

Table 8
Hydrogen-bond geometry (Å, °) for (VII).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O5^{i}	0.91 (1)	2.05 (1)	2.929 (4)	164 (3)
N1—H2N···O4^{ii}	0.90 (1)	2.17 (2)	2.980 (4)	149 (3)
N1—H2N···O3^{iii}	0.90 (1)	2.26 (3)	2.705 (3)	110 (2)
N1—H3N···O5	0.91 (1)	1.89 (1)	2.801 (3)	176 (3)
N2—H4N···N4^{iv}	0.89 (3)	1.99 (3)	2.882 (4)	174 (3)

Symmetry codes: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x+1, y, z$; (iii) $x+1, -y+\frac{1}{2}, z+\frac{1}{2}$; (iv) $-x+1, -y+1, -z+1$.

Table 9
Hydrogen-bond geometry (Å, °) for (VIII).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1*W*	0.95 (3)	1.80 (3)	2.733 (3)	170 (3)
N1—H2N···O2*W*^{i}	0.90 (3)	2.04 (3)	2.873 (3)	154 (3)
N1—H2N···O5^{ii}	0.90 (3)	2.32 (3)	2.793 (3)	113 (2)
N1—H3N···O6^{iii}	0.94 (3)	1.88 (3)	2.780 (3)	159 (3)
N2—H4N···N3^{iv}	0.87 (3)	2.03 (3)	2.897 (3)	179 (3)
O3—H1H···O2*W*	0.96 (4)	1.69 (4)	2.578 (3)	153 (4)
O1*W*—H1*W*···O3^{v}	0.87 (1)	1.89 (1)	2.760 (3)	173 (3)
O1*W*—H2*W*···O2^{vi}	0.87 (1)	1.96 (1)	2.834 (3)	175 (3)
O2*W*—H3*W*···O5^{vii}	0.88 (1)	1.85 (1)	2.722 (3)	172 (3)
O2*W*—H4*W*···O4^{ii}	0.88 (1)	1.88 (1)	2.737 (2)	163 (4)

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+\frac{1}{2}, y+\frac{3}{2}, -z+\frac{1}{2}$; (iv) $-x+1, -y+2, -z$; (v) $-x+1, -y+2, -z+1$; (vi) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (vii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

similar hydrogen-bonded solvent chains and instead form single hydrogen bonds to chloride. See Tables 2–9 for full details of the hydrogen bonding for compounds (I)–(VIII). Together, the water molecules and the chloride ions in (I) lie in sheets parallel to the *ac* plane. The V-shaped cations pack to give double layers and thus alternating layers of cations and solvent/halide ions exist along the crystallographic *b* direction (Fig. 3). Layer structures are seen for all the other structures herein.

Reactions with aqueous HBr and HI gave the bromide monohydrate, (III) (Fig. 4), and the tetraiodide, (IV) (Fig. 5). Both retain the layered structure described above, but perhaps, anti-intuitively, the hydrated structure with the simple bromide counter-ion is not isostructural with the chloride phases, whereas the anhydrous structure containing $[I_4]^{2-}$ is isostructural with the solvated chloride structures (Fig. 6), allowing only for a small increase in unit-cell size. This can again be shown by 20 out of 20 sulfadiazine cations having matching positions in a *Mercury* overlay. Although there are many species of polyiodide known, the $[I_4]^{2-}$ anion is a rare

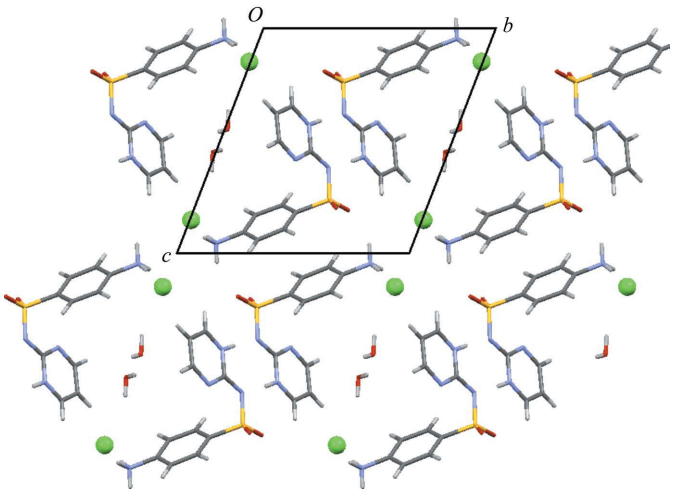

Figure 3
The packing of (I), viewed down the *a* axis. Note the bilayer of V-shaped cations separated by layers of chloride ions and water molecules. Here and in other figures, Cl = green, S = yellow, O = red and N = blue.

and thus much sought after variant (Weclawik *et al.*, 2013; Svensson & Kloo, 2003). Interestingly, a templating approach using a sulfonamide very closely related to sulfadiazine has been shown recently to allow access to tetraiodide species (Pan & Englert, 2014). In (IV), the $[I_4]^{2-}$ ion is nearly linear [$I2^{i}$—I2—I1 = 174.992 (16)°; symmetry code: (i) $-x-1, -y+1, -z+2$] and the I-to-I bonding is asymmetric, with a short central bond [I2—$I2^{i}$ = 2.7623 (6) Å] and two longer terminal bonds [I1—I2 = 3.3647 (4) Å]. Despite the large difference, both I-to-I distances are within the range routinely considered to denote bonding interactions and this is a fairly typical geometry for the $[I_4]^{2-}$ ion (Svensson & Kloo, 2003; Pan & Englert, 2014). The $[I_4]^{2-}$ ion lies on a crystallographic inversion centre; the similarity of the structure to the solvated chloride species is possible as the central I_2 unit occupies the

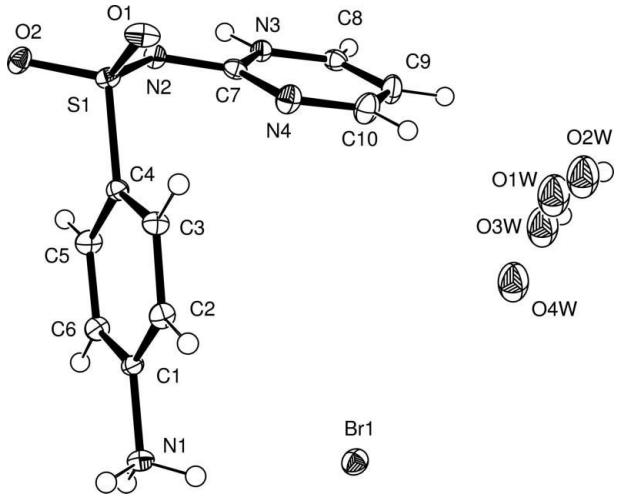

Figure 4
The molecular structure of (III), with non-H atoms shown as 50% probability displacement ellipsoids. O1*W*, O2*W*, O3*W* and O4*W* represent the partially occupied sites used to model a water molecule that is disordered in a channel parallel to the crystallographic *b* axis.

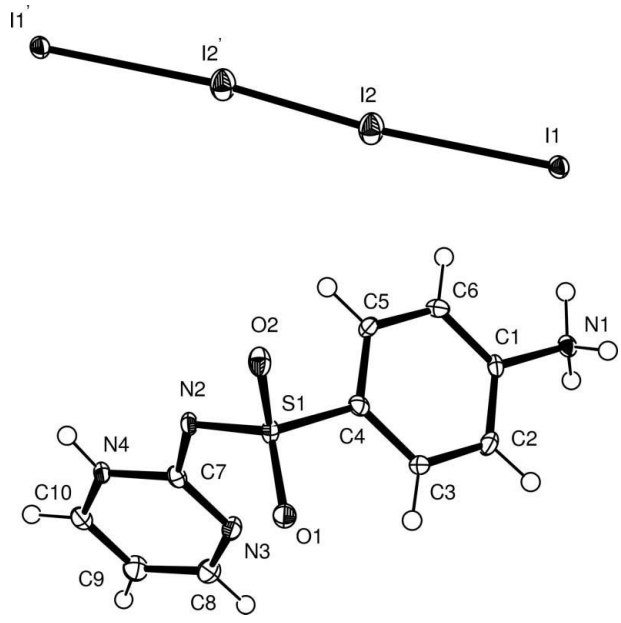

Figure 5
The molecular structure of (IV), with non-H atoms shown as 50% probability displacement ellipsoids. The contents of the asymmetric unit have been expanded to show a complete $[I_4]^{2-}$ ion. [Symmetry code: (i) $-x-1, -y+1, -z+2$.]

space taken by solvent in the chloride structures, whilst the terminal I atoms take up the positions occupied by the chloride ions. Note that the water molecules in the hydrobromide are disordered in channels parallel to the crystallographic *b* direction; such disorder is often seen for other channel hydrates. In bromide monohydrate (III), the water molecule has been modelled as split over four sites and these have been restrained to have a total occupancy of 1. H atoms

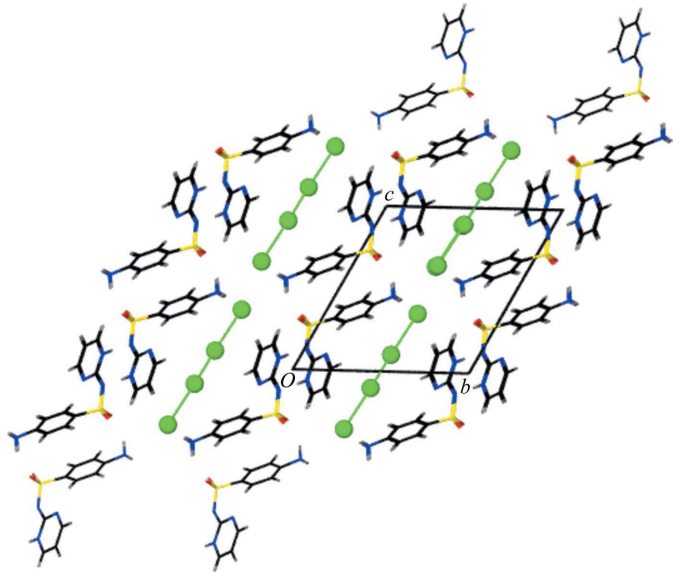

Figure 6
Packing diagram for (IV), viewed down the *a* axis. The central two I atoms of each I_4 unit occupy equivalent structural sites to the solvent molecules in the chloride solvate structures.

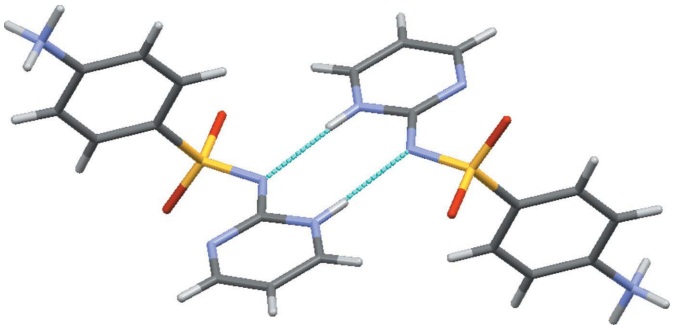

Figure 7
The centrosymmetric dimer which is robust enough to be found in all structures discussed herein. This example is drawn from structure (VI) and here the two cations are related by the symmetry operation $(-x+1, -y, -z+2)$.

could only be included for one of these sites, *i.e.* O1*W*, which is the water O-atom site with the largest occupancy [45.3 (10)%].

All these halide structures feature protonation at one of the pyrimidine ring N atoms and at the aniline group. This allows centrosymmetric hydrogen-bonded dimers to form which can be described using the $R_2^2(8)$ graph set (Bernstein *et al.*, 1995) (Fig. 7). For the ethylene glycol solvate of the hydrochloride salt, Pan *et al.* (2013) suggested that the unusual protonation of the aniline group was adopted as alternatives would disturb this energetically favourable dimeric interaction. This argument is strengthened by the persistence of the same dimeric unit through the halide structures presented here and indeed by the existence of the same dimeric unit in the hydrated tetrafluoroborate, (V) (Fig. 8), and nitrate, (VI) (Fig. 9), salt structures. These two structures also retain the fundamental layering structure of the halide species; compare Fig. 3 with Fig. 10. The $-NH_3^+$ group of all the halide species acts as a threefold hydrogen-bond donor. Two of the hydrogen bonds are to halide ions and thus link the cation layers to the anion layers, but the third hydrogen bond is formed with an O atom of the $-SO_2-$ fragment. This third hydrogen bond links the cationic dimers into a one-dimensional chain (Fig. 11). Nitrate

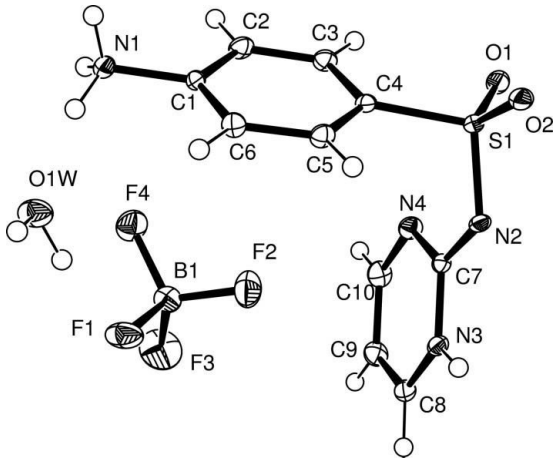

Figure 8
The molecular structure of (V), with non-H atoms shown as 50% probability displacement ellipsoids.

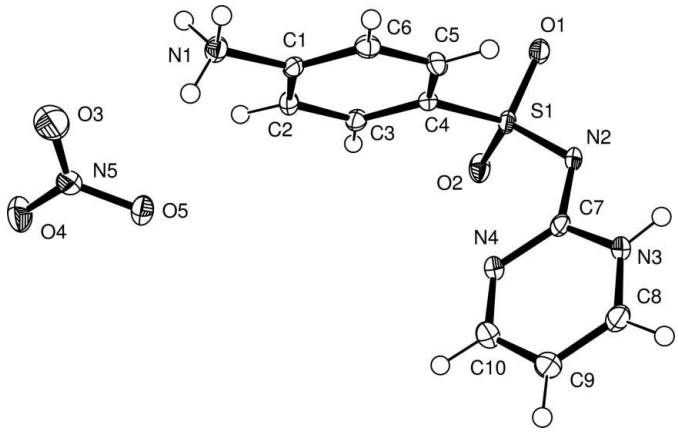

Figure 9
The molecular structure of (VI), with non-H atoms shown as 50% probability displacement ellipsoids.

structure (VI) is very similar, with the simple change that the $-NH_3^+$ group forms bifurcated hydrogen bonds with O atoms of the anions, whilst the structure of the BF_4^- species (V) differs slightly in that one $-NH_3^+$ H atom, *i.e.* H3N, forms a hydrogen bond with a water molecule acting as the acceptor. In all of structures (I)–(VI), this is the only direct sulfadiazine-to-solvent hydrogen bond.

The final two structures both have sulfonate-based anions, *viz.* ethanesulfonate in (VII) (Fig. 12) and 4-hydroxybenzenesulfonate in (VIII) (Fig. 13), and both are structurally different from the other species. In the sulfonate salts, the sulfadiazine is protonated at the aniline and amide N atoms and not at either of the pyrimidine ring N atoms. Despite this difference in tautomeric form, the $R_2^2(8)$ dimer of cations motif

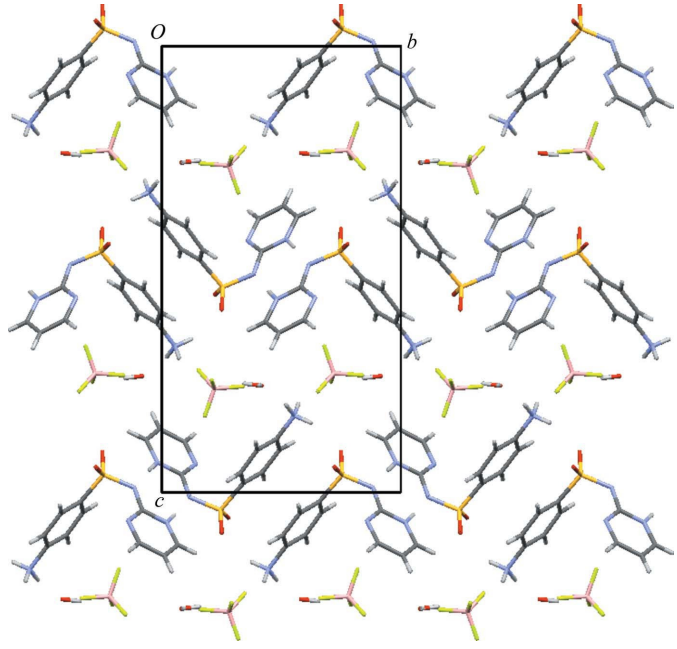

Figure 10
The packing of (V), viewed down the *a* axis. Although not part of the isostructural group formed by the chloride and $[I_4]^{2-}$ salts, the main stuctural features of bilayers of V-shaped cations separated by layers of anions and water molecules is retained.

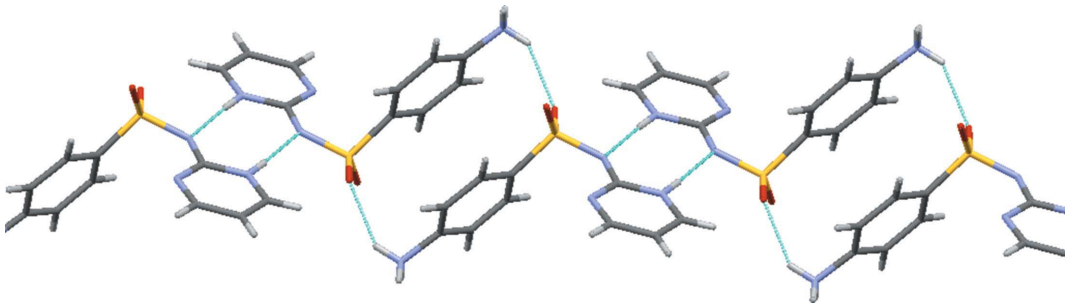

Figure 11
Part of the one-dimensional hydrogen-bonded chain of cations, drawn here for structure (II). The same supramolecular motif is found in structures (I)–(VI). Here the view is down the *a* axis and the chain extends along the [011] diagonal.

is retained. The dimeric structure is retained as the pyrimidine ring simply switches roles from hydrogen-bonding donor to hydrogen-bonding acceptor, whilst the amide group becomes the donor rather than the acceptor. Change to tautomeric form with retention of the same dimeric motif has also been described for related sulfonamides (Gelbrich *et al.*, 2007) and indeed can be seen when comparing the structure of neutral sulfadiazine with that of its salt forms (Pan *et al.*, 2013). In the latter case, change in tautomeric form was accompanied by change in molecular conformation. In particular, rotation about the S—C bond varied by approximately 35° between the amide-protonated species and the ring-protonated species. A similar division is not seen herein, with the dihedral angle between the planes defined by atoms N2/S1/C4 and the C1–C6 aromatic ring varying from 43.75 (10) to 64.13 (7)° for compounds (I)–(VI) and being 51.44 (14) and 60.12 (11)° for (VII) and (VIII), respectively. Elacqua *et al.* (2013) reported that neutral and anionic forms of sulfadiazine were differentiated by S—N bond distances, with neutral species having distances of 1.61–1.65 Å and anions having distances of 1.56–1.60 Å. The cationic sulfadiazines (I)–(VI) have distances intermediate to these values [range 1.5941 (17)–1.6061 (12) Å], whilst the amide-protonated species (VII) and (VIII) have longer bond lengths [1.633 (3) and 1.6422 (19) Å, respectively]. The adjacent N—C bonds are also slightly longer for (VII) and (VIII) [1.388 (4) and 1.386 (3) Å, respectively] than they are for the ring-protonated species (I)–(VI) [range 1.337 (3)–1.347 (5) Å]. Structures (VII) and (VIII) also differ from the other sulfadiazine salts as the $-SO_2-$ unit takes no part in hydrogen bonding other than with water and thus individual dimers do not connect through hydrogen bonding and so the polymeric motif described in Fig. 11 is absent. Instead, in ethanesulfonate salt (VII), the three H atoms of the $-NH_3^+$ group all donate hydrogen bonds to O atoms of the sulfonate group and in the 4-hydroxybenzenesulfonate dihydrate (VIII) the $-NH_3^+$ group acts as a hydrogen-bond donor to two water molecules and to one O atom of a sulfonate group. Although layered structures are retained, they are not the same as seen for the other species (Fig. 14). Only sulfonate-based anions appear to support sulfadiazine cations with the amide-protonated tautomeric form, but it is not obvious as to why this should be. As BF_4^- is a relatively poor hydrogen-bond acceptor, it may be that the tetrahedral shape of the sulfonate group together with its nature as a good and thus preferred hydrogen-bond acceptor is the key. However, whilst the structure of (VII), where ethanesulfonate forms multiple hydrogen-bonding interactions with the RNH_3^+ group of the sulfadiazine cation may support such a connection, the

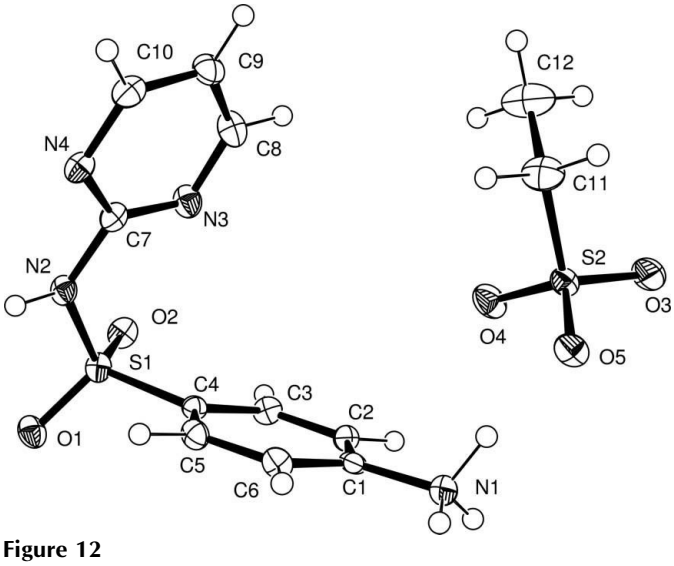

Figure 12
The molecular structure of (VII), with non-H atoms shown as 50% probability displacement ellipsoids.

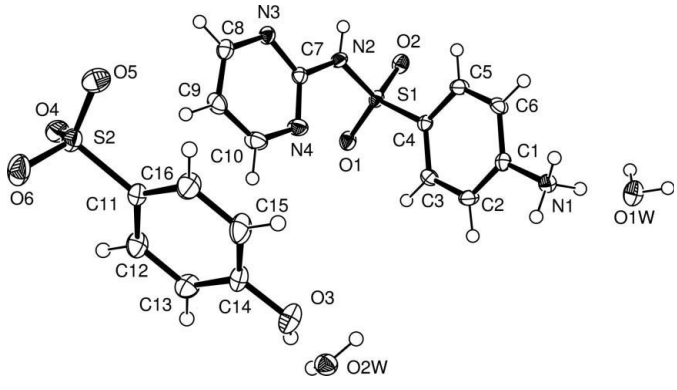

Figure 13
The molecular structure of (VIII), with non-H atoms shown as 50% probability displacement ellipsoids.

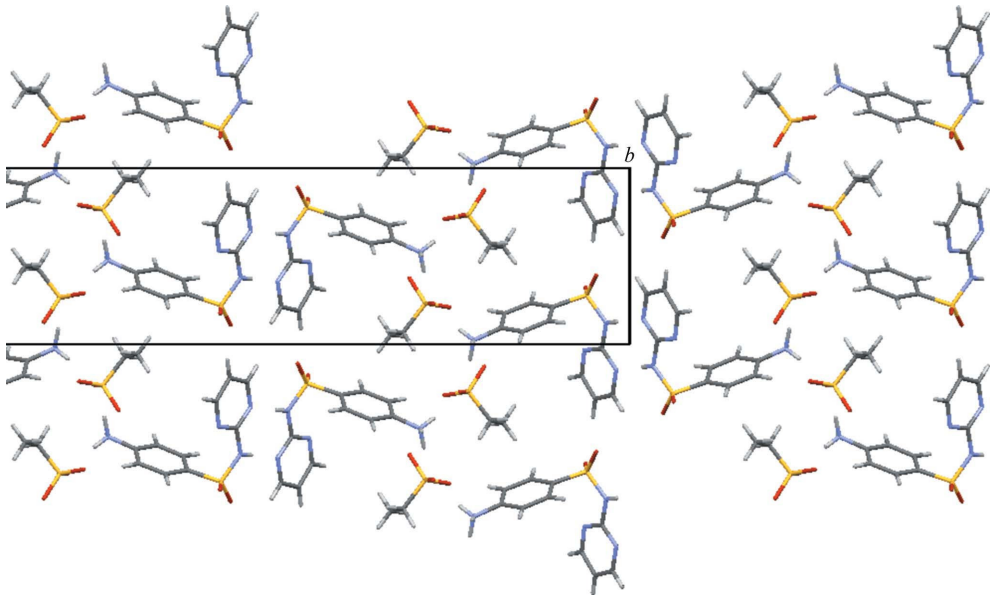

Figure 14
Packing diagram for (VII), viewed down the *a* axis, showing the alternating layer structure.

structure of dihydrate (VIII), where the sulfonate group mostly interacts with water molecules, does not.

We thank the National Crystallography Service (University of Southampton) for the data collections on (I) and (II) (Cole & Gale, 2012). CM wishes to thank the Nuffield Foundation for funding a research placement at the University of Strathclyde.

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supporting information

Acta Cryst. (2014). C**70**, 900-907 [doi:10.1107/S2053229614018725]

Eight salt forms of sulfadiazine

Amanda R. Buist, Lynn Dennany, Alan R. Kennedy, Craig Manzie, Katherine McPhie and Brandon Walker

Computing details

Data collection: *CrystalClear-SM Expert* (Rigaku, 2013) for (I), (II); *CrysAlis PRO* (Oxford Diffraction, 2010) for (III), (IV), (V), (VI), (VII), (VIII). Cell refinement: *CrystalClear-SM Expert* (Rigaku, 2013) for (I), (II); *CrysAlis PRO* (Oxford Diffraction, 2010) for (III), (IV), (V), (VI), (VII), (VIII). Data reduction: *CrystalClear-SM Expert* (Rigaku, 2013) for (I), (II); *CrysAlis PRO* (Oxford Diffraction, 2010) for (III), (IV), (V), (VI), (VII), (VIII). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994) for (I), (II); *SHELXS97* (Sheldrick, 2008) for (III), (IV), (V), (VI), (VII), (VIII). For all compounds, program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008). Molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008) for (I), (II), (V), (VI), (VII); *ORTEP-3 for Windows* (Farrugia, 2012) for (III), (VIII); *ORTEP-3 for Windows* (Farrugia, 2012) and *X-SEED* (Barbour, 2001) for (IV). For all compounds, software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

(I) 2-{[(4-Azaniumylphenyl)sulfonyl]azanidyl}pyrimidinium chloride monohydrate

Crystal data

$C_{10}H_{11}N_4O_2S^+\cdot Cl^-\cdot H_2O$
$M_r = 304.75$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 5.4118\ (4)$ Å
$b = 11.5632\ (8)$ Å
$c = 11.7430\ (8)$ Å
$\alpha = 109.462\ (7)°$
$\beta = 94.399\ (6)°$
$\gamma = 102.509\ (7)°$
$V = 667.51\ (8)$ Å^3
$Z = 2$
$F(000) = 316$
$D_x = 1.516$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 56264 reflections
$\theta = 3.1–27.5°$
$\mu = 0.45$ mm^{-1}
$T = 100$ K
Plate, yellow
0.06 × 0.02 × 0.01 mm

Data collection

Rigaku Saturn724+ (2x2 bin mode) diffractometer
Radiation source: fine-focus sealed tube
Mirrors monochromator
Detector resolution: 28.5714 pixels mm^{-1}
profile data from ω–scans
Absorption correction: multi-scan (*CrystalClear-SM Expert*; Rigaku, 2013)
$T_{min} = 0.897$, $T_{max} = 1.000$
8329 measured reflections
3000 independent reflections
2752 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -7\rightarrow7$
$k = -14\rightarrow14$
$l = -15\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.079$
$S = 1.05$
3000 reflections
197 parameters
5 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 0.4781P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

Special details

Experimental. NCS collection 2013ncs0582

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. One H atom of water molecule positioned as disordered over two sites.
Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl1	1.26745 (6)	1.00601 (3)	0.85165 (3)	0.01651 (10)	
S1	0.16642 (6)	0.43185 (3)	0.23791 (3)	0.01025 (9)	
O1	−0.07441 (19)	0.33435 (9)	0.19198 (9)	0.0140 (2)	
O2	0.3914 (2)	0.39099 (10)	0.20278 (9)	0.0155 (2)	
O1W	1.2540 (3)	0.98671 (16)	0.55066 (15)	0.0458 (4)	
H1W	1.277 (6)	1.013 (3)	0.6311 (9)	0.069*	
H2W	1.411 (4)	1.019 (5)	0.541 (4)	0.069*	0.50
H3W	1.122 (8)	1.017 (5)	0.540 (4)	0.069*	0.50
N1	0.1521 (2)	0.84971 (11)	0.04582 (11)	0.0129 (2)	
N2	0.1647 (2)	0.48996 (11)	0.38226 (10)	0.0119 (2)	
N3	0.3056 (2)	0.64473 (11)	0.57102 (11)	0.0117 (2)	
N4	0.5611 (2)	0.64491 (12)	0.41730 (11)	0.0146 (2)	
C1	0.1482 (3)	0.74771 (13)	0.09422 (12)	0.0111 (3)	
C2	0.3230 (3)	0.67568 (13)	0.06079 (12)	0.0128 (3)	
H2	0.4377	0.6913	0.0071	0.015*	
C3	0.3268 (3)	0.58038 (13)	0.10742 (12)	0.0122 (3)	
H3	0.4490	0.5318	0.0881	0.015*	
C4	0.1518 (3)	0.55606 (12)	0.18235 (12)	0.0101 (3)	
C5	−0.0265 (3)	0.62670 (13)	0.21399 (13)	0.0133 (3)	
H5	−0.1467	0.6083	0.2644	0.016*	
C6	−0.0256 (3)	0.72526 (13)	0.17032 (13)	0.0133 (3)	
H6	−0.1426	0.7764	0.1924	0.016*	

C7	0.3486 (3)	0.59269 (13)	0.45379 (12)	0.0111 (3)
C8	0.4704 (3)	0.74805 (13)	0.65421 (13)	0.0141 (3)
H8	0.4354	0.7823	0.7350	0.017*
C9	0.6903 (3)	0.80354 (14)	0.62089 (14)	0.0167 (3)
H9	0.8121	0.8765	0.6769	0.020*
C10	0.7250 (3)	0.74683 (14)	0.50033 (14)	0.0169 (3)
H10	0.8763	0.7835	0.4756	0.020*
H1N	0.310 (4)	0.906 (2)	0.070 (2)	0.029 (5)*
H2N	0.034 (4)	0.8936 (19)	0.0730 (18)	0.022 (5)*
H3N	0.118 (4)	0.814 (2)	−0.038 (2)	0.027 (5)*
H4N	0.162 (4)	0.6042 (19)	0.5895 (18)	0.023 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01210 (17)	0.01286 (17)	0.02310 (19)	0.00332 (12)	0.00203 (13)	0.00472 (13)
S1	0.01084 (17)	0.01154 (16)	0.00981 (16)	0.00382 (12)	0.00344 (11)	0.00467 (12)
O1	0.0147 (5)	0.0131 (5)	0.0124 (5)	0.0010 (4)	0.0021 (4)	0.0039 (4)
O2	0.0162 (5)	0.0198 (5)	0.0164 (5)	0.0102 (4)	0.0081 (4)	0.0095 (4)
O1W	0.0516 (10)	0.0450 (9)	0.0401 (8)	0.0006 (8)	0.0041 (7)	0.0226 (7)
N1	0.0132 (6)	0.0118 (6)	0.0143 (6)	0.0032 (5)	0.0023 (4)	0.0055 (5)
N2	0.0113 (5)	0.0143 (6)	0.0097 (5)	0.0021 (4)	0.0028 (4)	0.0043 (4)
N3	0.0108 (6)	0.0138 (5)	0.0115 (5)	0.0032 (4)	0.0033 (4)	0.0056 (4)
N4	0.0122 (6)	0.0174 (6)	0.0147 (6)	0.0023 (5)	0.0039 (4)	0.0070 (5)
C1	0.0113 (6)	0.0102 (6)	0.0104 (6)	0.0012 (5)	−0.0002 (5)	0.0035 (5)
C2	0.0116 (6)	0.0153 (6)	0.0120 (6)	0.0032 (5)	0.0041 (5)	0.0053 (5)
C3	0.0105 (6)	0.0143 (6)	0.0128 (6)	0.0054 (5)	0.0040 (5)	0.0045 (5)
C4	0.0099 (6)	0.0105 (6)	0.0096 (6)	0.0019 (5)	0.0010 (5)	0.0040 (5)
C5	0.0119 (6)	0.0158 (6)	0.0133 (6)	0.0038 (5)	0.0049 (5)	0.0057 (5)
C6	0.0118 (6)	0.0146 (6)	0.0146 (6)	0.0055 (5)	0.0039 (5)	0.0048 (5)
C7	0.0105 (6)	0.0131 (6)	0.0121 (6)	0.0049 (5)	0.0025 (5)	0.0064 (5)
C8	0.0166 (7)	0.0129 (6)	0.0131 (6)	0.0048 (5)	0.0018 (5)	0.0046 (5)
C9	0.0158 (7)	0.0139 (6)	0.0172 (7)	−0.0001 (5)	0.0002 (5)	0.0044 (5)
C10	0.0132 (7)	0.0178 (7)	0.0198 (7)	0.0010 (5)	0.0037 (5)	0.0087 (6)

Geometric parameters (Å, º)

S1—O2	1.4397 (10)	N4—C7	1.3484 (18)
S1—O1	1.4530 (10)	C1—C2	1.387 (2)
S1—N2	1.6034 (12)	C1—C6	1.3886 (19)
S1—C4	1.7777 (14)	C2—C3	1.387 (2)
O1W—H1W	0.880 (10)	C2—H2	0.9500
O1W—H2W	0.881 (10)	C3—C4	1.3886 (18)
O1W—H3W	0.882 (10)	C3—H3	0.9500
N1—C1	1.4671 (17)	C4—C5	1.3893 (19)
N1—H2N	0.91 (2)	C5—C6	1.396 (2)
N1—H3N	0.91 (2)	C5—H5	0.9500
N1—H1N	0.91 (2)	C6—H6	0.9500

N2—C7	1.3421 (18)	C8—C9	1.374 (2)
N3—C8	1.3431 (19)	C8—H8	0.9500
N3—C7	1.3670 (18)	C9—C10	1.394 (2)
N3—H4N	0.90 (2)	C9—H9	0.9500
N4—C10	1.3324 (19)	C10—H10	0.9500

O2—S1—O1	115.44 (6)	C3—C2—H2	120.7
O2—S1—N2	115.82 (6)	C2—C3—C4	119.88 (13)
O1—S1—N2	103.55 (6)	C2—C3—H3	120.1
O2—S1—C4	106.80 (6)	C4—C3—H3	120.1
O1—S1—C4	108.83 (6)	C3—C4—C5	121.48 (13)
N2—S1—C4	105.90 (6)	C3—C4—S1	117.44 (11)
H1W—O1W—H2W	99 (2)	C5—C4—S1	121.08 (10)
H1W—O1W—H3W	98 (2)	C4—C5—C6	118.77 (13)
H2W—O1W—H3W	125 (6)	C4—C5—H5	120.6
C1—N1—H2N	112.3 (13)	C6—C5—H5	120.6
C1—N1—H3N	108.3 (14)	C1—C6—C5	119.24 (13)
H2N—N1—H3N	109.1 (18)	C1—C6—H6	120.4
C1—N1—H1N	109.2 (14)	C5—C6—H6	120.4
H2N—N1—H1N	107.9 (18)	N2—C7—N4	124.87 (13)
H3N—N1—H1N	110.1 (18)	N2—C7—N3	114.71 (12)
C7—N2—S1	120.21 (10)	N4—C7—N3	120.42 (13)
C8—N3—C7	122.26 (12)	N3—C8—C9	119.12 (13)
C8—N3—H4N	122.0 (13)	N3—C8—H8	120.4
C7—N3—H4N	115.7 (13)	C9—C8—H8	120.4
C10—N4—C7	117.14 (13)	C8—C9—C10	116.37 (13)
C2—C1—C6	121.98 (13)	C8—C9—H9	121.8
C2—C1—N1	117.47 (12)	C10—C9—H9	121.8
C6—C1—N1	120.55 (12)	N4—C10—C9	124.70 (13)
C1—C2—C3	118.60 (12)	N4—C10—H10	117.7
C1—C2—H2	120.7	C9—C10—H10	117.7

O2—S1—N2—C7	59.45 (13)	S1—C4—C5—C6	178.43 (10)
O1—S1—N2—C7	−173.14 (11)	C2—C1—C6—C5	−0.5 (2)
C4—S1—N2—C7	−58.69 (12)	N1—C1—C6—C5	179.38 (12)
C6—C1—C2—C3	−1.5 (2)	C4—C5—C6—C1	1.7 (2)
N1—C1—C2—C3	178.65 (12)	S1—N2—C7—N4	−9.0 (2)
C1—C2—C3—C4	2.2 (2)	S1—N2—C7—N3	170.47 (10)
C2—C3—C4—C5	−1.1 (2)	C10—N4—C7—N2	−179.79 (13)
C2—C3—C4—S1	179.56 (10)	C10—N4—C7—N3	0.8 (2)
O2—S1—C4—C3	7.27 (13)	C8—N3—C7—N2	−179.80 (13)
O1—S1—C4—C3	−117.98 (11)	C8—N3—C7—N4	−0.3 (2)
N2—S1—C4—C3	131.25 (11)	C7—N3—C8—C9	−0.2 (2)
O2—S1—C4—C5	−172.08 (11)	N3—C8—C9—C10	0.2 (2)
O1—S1—C4—C5	62.68 (13)	C7—N4—C10—C9	−0.8 (2)
N2—S1—C4—C5	−48.09 (13)	C8—C9—C10—N4	0.3 (2)
C3—C4—C5—C6	−0.9 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···Cl1^{i}	0.91 (2)	2.27 (2)	3.1484 (13)	161.8 (19)
N1—H2*N*···Cl1^{ii}	0.91 (2)	2.27 (2)	3.1795 (13)	176.0 (18)
N1—H3*N*···O1^{iii}	0.91 (2)	1.99 (2)	2.8243 (16)	150.2 (19)
N3—H4*N*···N2^{iv}	0.90 (2)	1.98 (2)	2.8763 (17)	175.9 (19)
N3—H4*N*···O1^{iv}	0.90 (2)	2.54 (2)	3.0962 (15)	121.1 (16)
O1*W*—H1*W*···Cl1	0.88 (1)	2.62 (1)	3.4607 (17)	160 (3)
O1*W*—H2*W*···O1*W*^{v}	0.88 (1)	2.18 (2)	2.995 (4)	153 (4)
O1*W*—H3*W*···O1*W*^{i}	0.88 (1)	2.21 (2)	3.030 (4)	155 (5)

Symmetry codes: (i) −*x*+2, −*y*+2, −*z*+1; (ii) −*x*+1, −*y*+2, −*z*+1; (iii) −*x*, −*y*+1, −*z*; (iv) −*x*, −*y*+1, −*z*+1; (v) −*x*+3, −*y*+2, −*z*+1.

(II) 2-{[(4-azaniumylphenyl)sulfonyl]azanidyl}pyrimidin-1-ium chloride methanol hemisolvate

Crystal data

$C_{10}H_{11}N_4O_2S^+\cdot Cl^-\cdot 0.5CH_4O$
M_r = 302.76
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 5.6468 (2) Å
b = 11.2749 (4) Å
c = 11.8363 (8) Å
α = 64.254 (5)°
β = 79.361 (6)°
γ = 77.687 (6)°
V = 659.53 (6) Å^3
Z = 2
$F(000)$ = 314
D_x = 1.525 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 10881 reflections
θ = 3.5–27.5°
μ = 0.45 mm^{-1}
T = 100 K
Block, light yellow
0.09 × 0.06 × 0.05 mm

Data collection

Rigaku Saturn724+ (2x2 bin mode) diffractometer
Radiation source: Rotating Anode
Confocal monochromator
profile data from ω–scans
Absorption correction: multi-scan (*CrystalClear-SM Expert*; Rigaku, 2013)
T_{min} = 0.819, T_{max} = 1.000
11731 measured reflections
3028 independent reflections
2804 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 27.5°, θ_{min} = 3.3°
h = −7→7
k = −14→14
l = −15→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.082
S = 1.04
3028 reflections
201 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0374P)^2 + 0.5228P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

Special details

Experimental. Collected by NCS as 2013ncs0086.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Methanol disordered about i.

Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.40767 (6)	0.72559 (3)	0.22516 (3)	0.01372 (10)	
Cl1	0.75329 (6)	0.38116 (4)	0.14375 (3)	0.01880 (10)	
O1	0.65919 (19)	0.68725 (10)	0.18505 (10)	0.0176 (2)	
O2	0.2257 (2)	0.70618 (10)	0.16657 (10)	0.0186 (2)	
N1	0.3079 (2)	1.31394 (12)	0.05936 (13)	0.0161 (3)	
N2	0.3892 (2)	0.64417 (12)	0.37570 (11)	0.0150 (2)	
N3	0.1813 (2)	0.55649 (12)	0.57023 (12)	0.0150 (2)	
N4	−0.0209 (2)	0.74149 (13)	0.41155 (12)	0.0183 (3)	
C7	0.1794 (3)	0.65129 (14)	0.44874 (13)	0.0147 (3)	
C8	−0.0120 (3)	0.54958 (15)	0.65718 (14)	0.0172 (3)	
H8	−0.0051	0.4833	0.7410	0.021*	
C9	−0.2192 (3)	0.63983 (16)	0.62264 (15)	0.0195 (3)	
H9	−0.3603	0.6383	0.6808	0.023*	
C10	−0.2123 (3)	0.73386 (16)	0.49799 (15)	0.0204 (3)	
H10	−0.3544	0.7972	0.4727	0.024*	
C4	0.3707 (3)	0.89860 (14)	0.18623 (13)	0.0143 (3)	
C3	0.5466 (3)	0.95072 (15)	0.21162 (14)	0.0167 (3)	
H3	0.6812	0.8934	0.2547	0.020*	
C2	0.5231 (3)	1.08797 (15)	0.17307 (14)	0.0168 (3)	
H2	0.6386	1.1253	0.1917	0.020*	
C1	0.3284 (3)	1.16919 (14)	0.10703 (13)	0.0144 (3)	
C6	0.1533 (3)	1.11789 (14)	0.08048 (14)	0.0160 (3)	
H6	0.0221	1.1755	0.0346	0.019*	
C5	0.1735 (3)	0.98098 (15)	0.12211 (14)	0.0159 (3)	
H5	0.0534	0.9436	0.1069	0.019*	
O3	0.7639 (6)	0.1624 (4)	0.4251 (3)	0.0507 (8)	0.50
H1H	0.794 (12)	0.216 (5)	0.345 (2)	0.076*	0.50
C11	0.9255 (11)	0.0552 (5)	0.4402 (5)	0.0568 (13)	0.50
H11A	1.0887	0.0758	0.4339	0.085*	0.50
H11B	0.9192	0.0253	0.3746	0.085*	0.50
H11C	0.8889	−0.0155	0.5234	0.085*	0.50
H1N	0.428 (4)	1.339 (2)	0.0828 (19)	0.024 (5)*	
H2N	0.319 (4)	1.351 (2)	−0.022 (2)	0.033 (6)*	

H3N	0.156 (5)	1.349 (2)	0.085 (2)	0.038 (6)*
H4N	0.316 (4)	0.497 (2)	0.5888 (19)	0.027 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01316 (17)	0.01214 (17)	0.01348 (17)	−0.00231 (12)	−0.00062 (12)	−0.00321 (13)
Cl1	0.01368 (17)	0.01857 (18)	0.02504 (19)	−0.00376 (13)	0.00072 (13)	−0.01025 (15)
O1	0.0154 (5)	0.0164 (5)	0.0167 (5)	−0.0003 (4)	0.0013 (4)	−0.0050 (4)
O2	0.0206 (5)	0.0163 (5)	0.0185 (5)	−0.0053 (4)	−0.0052 (4)	−0.0044 (4)
N1	0.0145 (6)	0.0136 (6)	0.0184 (6)	−0.0026 (5)	−0.0008 (5)	−0.0050 (5)
N2	0.0131 (6)	0.0139 (6)	0.0142 (6)	−0.0025 (4)	0.0002 (4)	−0.0026 (5)
N3	0.0134 (6)	0.0140 (6)	0.0151 (6)	−0.0021 (5)	−0.0011 (5)	−0.0039 (5)
N4	0.0148 (6)	0.0181 (6)	0.0182 (6)	−0.0011 (5)	−0.0015 (5)	−0.0047 (5)
C7	0.0138 (6)	0.0135 (6)	0.0165 (7)	−0.0034 (5)	−0.0017 (5)	−0.0054 (5)
C8	0.0167 (7)	0.0175 (7)	0.0167 (7)	−0.0060 (5)	0.0011 (5)	−0.0061 (6)
C9	0.0139 (7)	0.0234 (7)	0.0203 (7)	−0.0038 (6)	0.0017 (5)	−0.0090 (6)
C10	0.0141 (7)	0.0216 (7)	0.0227 (8)	0.0001 (6)	−0.0022 (6)	−0.0077 (6)
C4	0.0133 (6)	0.0127 (6)	0.0141 (6)	−0.0026 (5)	0.0007 (5)	−0.0035 (5)
C3	0.0137 (6)	0.0166 (7)	0.0175 (7)	−0.0019 (5)	−0.0035 (5)	−0.0043 (5)
C2	0.0141 (6)	0.0172 (7)	0.0187 (7)	−0.0046 (5)	−0.0024 (5)	−0.0058 (6)
C1	0.0141 (6)	0.0125 (6)	0.0143 (6)	−0.0025 (5)	0.0015 (5)	−0.0042 (5)
C6	0.0129 (6)	0.0158 (7)	0.0172 (7)	−0.0010 (5)	−0.0026 (5)	−0.0050 (5)
C5	0.0130 (6)	0.0166 (7)	0.0168 (7)	−0.0033 (5)	−0.0016 (5)	−0.0053 (5)
O3	0.0399 (17)	0.062 (2)	0.0376 (17)	−0.0020 (15)	−0.0035 (14)	−0.0118 (16)
C11	0.073 (4)	0.034 (2)	0.064 (3)	−0.013 (2)	−0.003 (3)	−0.020 (2)

Geometric parameters (Å, º)

S1—O2	1.4407 (11)	C9—H9	0.9500
S1—O1	1.4481 (11)	C10—H10	0.9500
S1—N2	1.6061 (12)	C4—C3	1.391 (2)
S1—C4	1.7725 (14)	C4—C5	1.392 (2)
N1—C1	1.4625 (18)	C3—C2	1.394 (2)
N1—H1N	0.91 (2)	C3—H3	0.9500
N1—H2N	0.86 (2)	C2—C1	1.387 (2)
N1—H3N	0.92 (3)	C2—H2	0.9500
N2—C7	1.3413 (19)	C1—C6	1.388 (2)
N3—C8	1.3449 (19)	C6—C5	1.387 (2)
N3—C7	1.3697 (19)	C6—H6	0.9500
N3—H4N	0.89 (2)	C5—H5	0.9500
N4—C10	1.333 (2)	O3—C11	1.314 (6)
N4—C7	1.3461 (19)	O3—H1H	0.886 (10)
C8—C9	1.371 (2)	C11—H11A	0.9800
C8—H8	0.9500	C11—H11B	0.9800
C9—C10	1.393 (2)	C11—H11C	0.9800
O2—S1—O1	116.27 (7)	C8—C9—H9	121.7

O2—S1—N2	113.94 (7)	C10—C9—H9	121.7
O1—S1—N2	103.29 (6)	N4—C10—C9	124.82 (14)
O2—S1—C4	106.85 (7)	N4—C10—H10	117.6
O1—S1—C4	106.51 (7)	C9—C10—H10	117.6
N2—S1—C4	109.69 (7)	C3—C4—C5	121.26 (13)
C1—N1—H1N	112.2 (13)	C3—C4—S1	119.87 (11)
C1—N1—H2N	111.2 (15)	C5—C4—S1	118.70 (11)
H1N—N1—H2N	106.9 (19)	C4—C3—C2	119.21 (13)
C1—N1—H3N	109.9 (15)	C4—C3—H3	120.4
H1N—N1—H3N	111.3 (19)	C2—C3—H3	120.4
H2N—N1—H3N	105 (2)	C1—C2—C3	118.95 (13)
C7—N2—S1	121.68 (10)	C1—C2—H2	120.5
C8—N3—C7	122.36 (13)	C3—C2—H2	120.5
C8—N3—H4N	120.9 (14)	C2—C1—C6	122.10 (13)
C7—N3—H4N	116.7 (14)	C2—C1—N1	119.86 (13)
C10—N4—C7	116.95 (13)	C6—C1—N1	118.01 (13)
N2—C7—N4	125.45 (13)	C5—C6—C1	118.78 (13)
N2—C7—N3	114.12 (13)	C5—C6—H6	120.6
N4—C7—N3	120.43 (13)	C1—C6—H6	120.6
N3—C8—C9	118.82 (14)	C6—C5—C4	119.67 (14)
N3—C8—H8	120.6	C6—C5—H5	120.2
C9—C8—H8	120.6	C4—C5—H5	120.2
C8—C9—C10	116.63 (14)	C11—O3—H1H	104 (4)

O2—S1—N2—C7	−51.72 (14)	N2—S1—C4—C3	66.90 (13)
O1—S1—N2—C7	−178.76 (12)	O2—S1—C4—C5	6.20 (13)
C4—S1—N2—C7	68.00 (13)	O1—S1—C4—C5	131.09 (12)
S1—N2—C7—N4	−12.9 (2)	N2—S1—C4—C5	−117.77 (12)
S1—N2—C7—N3	167.34 (10)	C5—C4—C3—C2	0.6 (2)
C10—N4—C7—N2	−179.34 (14)	S1—C4—C3—C2	175.85 (11)
C10—N4—C7—N3	0.4 (2)	C4—C3—C2—C1	−1.9 (2)
C8—N3—C7—N2	179.11 (13)	C3—C2—C1—C6	1.4 (2)
C8—N3—C7—N4	−0.6 (2)	C3—C2—C1—N1	−176.48 (13)
C7—N3—C8—C9	0.6 (2)	C2—C1—C6—C5	0.4 (2)
N3—C8—C9—C10	−0.4 (2)	N1—C1—C6—C5	178.33 (13)
C7—N4—C10—C9	−0.2 (2)	C1—C6—C5—C4	−1.7 (2)
C8—C9—C10—N4	0.2 (2)	C3—C4—C5—C6	1.2 (2)
O2—S1—C4—C3	−169.13 (11)	S1—C4—C5—C6	−174.09 (11)
O1—S1—C4—C3	−44.24 (13)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···Cl1^{i}	0.91 (2)	2.30 (2)	3.2088 (14)	174.1 (18)
N1—H2*N*···Cl1^{ii}	0.86 (2)	2.70 (2)	3.2151 (13)	119.8 (18)
N1—H2*N*···O1^{ii}	0.86 (2)	2.13 (2)	2.8701 (18)	144 (2)
N1—H3*N*···Cl1^{iii}	0.92 (3)	2.25 (3)	3.1417 (14)	163 (2)
N3—H4*N*···N2^{iv}	0.89 (2)	2.01 (2)	2.8957 (18)	175.4 (19)

N3—H4*N*···O1^{iv}	0.89 (2)	2.59 (2)	3.1218 (16)	119.6 (16)
O3—H1*H*···Cl1	0.89 (1)	2.31 (2)	3.170 (3)	163 (6)

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) −*x*+1, −*y*+2, −*z*; (iii) *x*−1, *y*+1, *z*; (iv) −*x*+1, −*y*+1, −*z*+1.

(III) 2-{[(4-Azaniumylphenyl)sulfonyl]azanidyl}pyrimidin-1-ium bromide monohydrate

Crystal data

$C_{10}H_{11}N_4O_2S^+·Br^-·H_2O$
M_r = 349.21
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 11.9420 (3) Å
b = 5.6295 (2) Å
c = 21.0659 (6) Å
β = 98.380 (3)°
V = 1401.09 (7) Å^3
Z = 4
$F(000)$ = 704
D_x = 1.656 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4492 reflections
θ = 3.2–29.8°
μ = 3.09 mm^{-1}
T = 123 K
Plate, colourless
0.30 × 0.22 × 0.04 mm

Data collection

Oxford Diffraction Xcalibur E diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.616, T_{max} = 1.000
8186 measured reflections
3353 independent reflections
2868 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 28.0°, θ_{min} = 3.7°
h = −15→15
k = −6→7
l = −25→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.072
S = 1.06
3353 reflections
203 parameters
5 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2 + 0.9997P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlisPro, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Br1	0.401273 (18)	0.49939 (4)	0.747817 (10)	0.01647 (8)	
S1	0.76326 (4)	0.82481 (10)	1.03211 (2)	0.01199 (12)	
O1	0.74525 (12)	0.5931 (3)	1.05768 (8)	0.0183 (3)	
O2	0.76800 (13)	1.0205 (3)	1.07703 (8)	0.0162 (3)	
N1	0.37146 (16)	0.9984 (4)	0.82655 (9)	0.0137 (4)	
H1N	0.360 (2)	0.870 (5)	0.8004 (13)	0.021*	
H2N	0.310 (3)	1.006 (4)	0.8447 (14)	0.021*	
H3N	0.378 (2)	1.127 (5)	0.8058 (13)	0.021*	
N2	0.87783 (14)	0.8530 (3)	1.00155 (9)	0.0131 (4)	
N3	0.99972 (14)	0.7555 (3)	0.93244 (9)	0.0129 (4)	
H4N	1.0374 (18)	0.871 (3)	0.9527 (11)	0.015*	
N4	0.83362 (16)	0.5297 (3)	0.92931 (10)	0.0181 (4)	
C4	0.64922 (16)	0.8809 (4)	0.97023 (10)	0.0118 (4)	
C3	0.56750 (17)	0.7068 (4)	0.95621 (10)	0.0131 (4)	
H3	0.5746	0.5596	0.9785	0.016*	
C2	0.47470 (17)	0.7483 (4)	0.90921 (10)	0.0138 (4)	
H2	0.4164	0.6332	0.9002	0.017*	
C1	0.46949 (17)	0.9603 (4)	0.87620 (10)	0.0116 (4)	
C6	0.55169 (17)	1.1346 (4)	0.88907 (10)	0.0146 (4)	
H6	0.5464	1.2783	0.8652	0.017*	
C5	0.64198 (18)	1.0956 (4)	0.93754 (11)	0.0153 (4)	
H5	0.6980	1.2144	0.9482	0.018*	
C7	0.90044 (17)	0.7083 (4)	0.95472 (10)	0.0129 (4)	
C10	0.86942 (19)	0.4047 (5)	0.88206 (12)	0.0217 (5)	
H10	0.8229	0.2780	0.8638	0.026*	
C9	0.9693 (2)	0.4467 (4)	0.85792 (12)	0.0201 (5)	
H9	0.9912	0.3532	0.8243	0.024*	
C8	1.03513 (18)	0.6303 (4)	0.88507 (10)	0.0156 (5)	
H8	1.1047	0.6680	0.8705	0.019*	
O1W	0.8026 (5)	0.0239 (18)	0.7626 (4)	0.0386 (16)	0.453 (10)
H1W	0.826 (8)	−0.122 (6)	0.759 (5)	0.058*	0.45
H2W	0.830 (9)	0.088 (16)	0.730 (4)	0.058*	0.45
O2W	0.8094 (12)	−0.082 (3)	0.7675 (8)	0.0386 (16)	0.248 (10)
O3W	0.8131 (13)	0.141 (3)	0.7321 (9)	0.0386 (16)	0.161 (6)
O4W	0.7780 (11)	0.300 (3)	0.7019 (8)	0.0386 (16)	0.139 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.01972 (12)	0.01516 (13)	0.01408 (12)	−0.00096 (8)	0.00096 (8)	0.00103 (8)
S1	0.0089 (2)	0.0151 (3)	0.0120 (2)	−0.0023 (2)	0.00132 (17)	0.0010 (2)
O1	0.0135 (7)	0.0192 (8)	0.0213 (9)	−0.0033 (6)	−0.0004 (6)	0.0081 (7)
O2	0.0128 (7)	0.0225 (9)	0.0138 (8)	−0.0027 (6)	0.0033 (6)	−0.0046 (6)
N1	0.0112 (8)	0.0162 (10)	0.0131 (9)	−0.0013 (7)	0.0000 (7)	0.0021 (8)
N2	0.0093 (8)	0.0150 (10)	0.0154 (9)	−0.0025 (7)	0.0033 (6)	−0.0017 (7)

N3	0.0105 (8)	0.0136 (9)	0.0145 (9)	−0.0018 (7)	0.0017 (6)	−0.0008 (7)
N4	0.0156 (9)	0.0180 (11)	0.0208 (10)	−0.0057 (7)	0.0033 (7)	−0.0036 (8)
C4	0.0098 (9)	0.0141 (11)	0.0117 (10)	0.0000 (8)	0.0021 (7)	0.0001 (8)
C3	0.0135 (10)	0.0116 (11)	0.0141 (10)	−0.0018 (8)	0.0017 (8)	0.0035 (8)
C2	0.0116 (9)	0.0127 (11)	0.0170 (11)	−0.0043 (8)	0.0024 (8)	−0.0005 (8)
C1	0.0093 (9)	0.0162 (11)	0.0094 (10)	0.0005 (8)	0.0014 (7)	−0.0004 (8)
C6	0.0168 (10)	0.0118 (11)	0.0149 (11)	−0.0016 (8)	0.0019 (8)	0.0018 (8)
C5	0.0140 (10)	0.0129 (11)	0.0185 (11)	−0.0052 (8)	0.0008 (8)	0.0010 (9)
C7	0.0115 (9)	0.0119 (11)	0.0151 (11)	−0.0001 (8)	0.0011 (7)	0.0029 (8)
C10	0.0209 (11)	0.0205 (12)	0.0235 (13)	−0.0062 (10)	0.0027 (9)	−0.0061 (10)
C9	0.0219 (11)	0.0197 (13)	0.0194 (12)	0.0007 (9)	0.0052 (9)	−0.0070 (9)
C8	0.0127 (10)	0.0188 (12)	0.0155 (11)	0.0025 (8)	0.0030 (8)	0.0021 (9)
O1W	0.0258 (17)	0.037 (5)	0.053 (3)	−0.003 (3)	0.0057 (17)	−0.018 (3)
O2W	0.0258 (17)	0.037 (5)	0.053 (3)	−0.003 (3)	0.0057 (17)	−0.018 (3)
O3W	0.0258 (17)	0.037 (5)	0.053 (3)	−0.003 (3)	0.0057 (17)	−0.018 (3)
O4W	0.0258 (17)	0.037 (5)	0.053 (3)	−0.003 (3)	0.0057 (17)	−0.018 (3)

Geometric parameters (Å, º)

S1—O1	1.4394 (17)	C2—C1	1.378 (3)
S1—O2	1.4480 (16)	C2—H2	0.9500
S1—N2	1.6026 (18)	C1—C6	1.387 (3)
S1—C4	1.771 (2)	C6—C5	1.390 (3)
N1—C1	1.468 (3)	C6—H6	0.9500
N1—H1N	0.91 (3)	C5—H5	0.9500
N1—H2N	0.88 (3)	C10—C9	1.384 (3)
N1—H3N	0.85 (3)	C10—H10	0.9500
N2—C7	1.337 (3)	C9—C8	1.372 (3)
N3—C8	1.340 (3)	C9—H9	0.9500
N3—C7	1.363 (3)	C8—H8	0.9500
N3—H4N	0.869 (10)	O1W—H1W	0.876 (10)
N4—C10	1.339 (3)	O1W—H2W	0.877 (10)
N4—C7	1.345 (3)	O2W—O3W	1.46 (2)
C4—C3	1.384 (3)	O2W—O4W^{i}	1.46 (2)
C4—C5	1.387 (3)	O3W—O4W	1.14 (2)
C3—C2	1.394 (3)	O4W—O2W^{ii}	1.46 (2)
C3—H3	0.9500		

O1—S1—O2	116.03 (10)	C1—C2—H2	120.8
O1—S1—N2	115.02 (10)	C3—C2—H2	120.8
O2—S1—N2	103.46 (9)	C2—C1—C6	122.39 (19)
O1—S1—C4	107.23 (10)	C2—C1—N1	117.33 (18)
O2—S1—C4	107.34 (10)	C6—C1—N1	120.27 (19)
N2—S1—C4	107.26 (10)	C1—C6—C5	118.9 (2)
C1—N1—H1N	110.5 (16)	C1—C6—H6	120.6
C1—N1—H2N	109.2 (19)	C5—C6—H6	120.6
H1N—N1—H2N	104 (2)	C4—C5—C6	119.2 (2)
C1—N1—H3N	111.6 (18)	C4—C5—H5	120.4

H1N—N1—H3N	113 (3)	C6—C5—H5	120.4
H2N—N1—H3N	109 (2)	N2—C7—N4	125.2 (2)
C7—N2—S1	120.81 (15)	N2—C7—N3	114.51 (18)
C8—N3—C7	122.61 (19)	N4—C7—N3	120.2 (2)
C8—N3—H4N	124.3 (17)	N4—C10—C9	124.7 (2)
C7—N3—H4N	113.1 (17)	N4—C10—H10	117.7
C10—N4—C7	117.0 (2)	C9—C10—H10	117.7
C3—C4—C5	121.32 (18)	C8—C9—C10	116.6 (2)
C3—C4—S1	118.25 (16)	C8—C9—H9	121.7
C5—C4—S1	120.42 (15)	C10—C9—H9	121.7
C4—C3—C2	119.7 (2)	N3—C8—C9	118.9 (2)
C4—C3—H3	120.2	N3—C8—H8	120.6
C2—C3—H3	120.2	C9—C8—H8	120.6
C1—C2—C3	118.46 (19)	H1W—O1W—H2W	99 (2)
O1—S1—N2—C7	55.99 (19)	N1—C1—C6—C5	178.3 (2)
O2—S1—N2—C7	−176.45 (16)	C3—C4—C5—C6	−1.0 (3)
C4—S1—N2—C7	−63.17 (19)	S1—C4—C5—C6	179.84 (18)
O1—S1—C4—C3	−0.9 (2)	C1—C6—C5—C4	2.0 (3)
O2—S1—C4—C3	−126.17 (18)	S1—N2—C7—N4	−0.6 (3)
N2—S1—C4—C3	123.19 (18)	S1—N2—C7—N3	178.80 (14)
O1—S1—C4—C5	178.30 (18)	C10—N4—C7—N2	179.2 (2)
O2—S1—C4—C5	53.0 (2)	C10—N4—C7—N3	−0.1 (3)
N2—S1—C4—C5	−57.6 (2)	C8—N3—C7—N2	−179.16 (19)
C5—C4—C3—C2	−1.2 (3)	C8—N3—C7—N4	0.2 (3)
S1—C4—C3—C2	177.92 (17)	C7—N4—C10—C9	−0.1 (4)
C4—C3—C2—C1	2.4 (3)	N4—C10—C9—C8	0.1 (4)
C3—C2—C1—C6	−1.5 (3)	C7—N3—C8—C9	−0.2 (3)
C3—C2—C1—N1	179.4 (2)	C10—C9—C8—N3	0.1 (3)
C2—C1—C6—C5	−0.8 (3)		

Symmetry codes: (i) −*x*+3/2, *y*−1/2, −*z*+3/2; (ii) −*x*+3/2, *y*+1/2, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···Br1	0.91 (3)	2.44 (3)	3.308 (2)	159 (2)
N1—H2*N*···O2^{iii}	0.88 (3)	2.01 (3)	2.810 (3)	150 (3)
N1—H2*N*···Br1^{iv}	0.88 (3)	2.95 (3)	3.4022 (18)	114 (2)
N1—H3*N*···Br1^{v}	0.85 (3)	2.46 (3)	3.317 (2)	178 (2)
N3—H4*N*···N2^{vi}	0.87 (1)	2.02 (1)	2.887 (2)	179 (2)
N3—H4*N*···O2^{vi}	0.87 (1)	2.57 (2)	3.080 (2)	119 (2)

Symmetry codes: (iii) −*x*+1, −*y*+2, −*z*+2; (iv) −*x*+1/2, *y*+1/2, −*z*+3/2; (v) *x*, *y*+1, *z*; (vi) −*x*+2, −*y*+2, −*z*+2.

(IV) Bis(2-{[(4-azaniumylphenyl)sulfonyl]azanidyl}pyrimidin-1-ium) tetraiodide

Crystal data

$2C_{10}H_{11}N_4O_2S^+·I_4^{2-}$
M_r = 1010.18
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 5.7792 (4) Å
b = 12.0002 (7) Å
c = 12.5214 (9) Å
α = 61.964 (7)°
β = 87.745 (5)°
γ = 76.426 (5)°
V = 742.51 (9) Å^3
Z = 1
$F(000)$ = 474
D_x = 2.259 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3604 reflections
θ = 3.3–29.4°
μ = 4.38 mm^{-1}
T = 123 K
Plate, red
0.25 × 0.15 × 0.02 mm

Data collection

Oxford Diffraction Xcalibur E diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: analytical [*CrysAlis PRO* (Oxford Diffraction, 2010), based on expressions derived by Clark & Reid (1995)]
T_{min} = 0.523, T_{max} = 0.905
7055 measured reflections
3595 independent reflections
2996 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 29.4°, θ_{min} = 3.3°
h = −7→7
k = −16→15
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.062
S = 1.04
3595 reflections
184 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0159P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.93 e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlisPro, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40) Analytical numeric absorption correction using a multifaceted crystal model based on expressions derived by R.C. Clark & J.S. Reid. (Clark, R. C. & Reid, J. S. (1995). Acta Cryst. A51, 887-897)

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	−0.24113 (4)	0.47686 (3)	0.64525 (2)	0.01501 (8)
I2	−0.41230 (5)	0.49574 (3)	0.89742 (3)	0.02602 (9)
S1	0.10433 (17)	1.04954 (10)	0.73009 (8)	0.0122 (2)
O1	0.2872 (5)	1.1182 (3)	0.6759 (2)	0.0165 (6)
O2	−0.1411 (5)	1.1268 (3)	0.6924 (2)	0.0157 (6)
N1	0.1784 (6)	0.6688 (4)	0.5445 (3)	0.0127 (7)
H1N	0.317 (7)	0.617 (4)	0.567 (4)	0.015*
H2N	0.169 (7)	0.714 (4)	0.468 (4)	0.015*
H3N	0.042 (7)	0.632 (4)	0.563 (3)	0.015*
N2	0.1152 (5)	0.9858 (3)	0.8748 (3)	0.0117 (7)
N3	0.5180 (5)	0.8604 (3)	0.9033 (3)	0.0140 (7)
N4	0.3154 (6)	0.8837 (3)	1.0623 (3)	0.0122 (7)
H4N	0.175 (7)	0.920 (4)	1.083 (3)	0.015*
C1	0.1645 (6)	0.7559 (4)	0.5968 (3)	0.0111 (8)
C2	0.3396 (7)	0.8239 (4)	0.5738 (3)	0.0142 (8)
H2	0.4666	0.8123	0.5265	0.017*
C3	0.3271 (7)	0.9083 (4)	0.6202 (3)	0.0128 (8)
H3	0.4488	0.9533	0.6080	0.015*
C4	0.1344 (6)	0.9274 (4)	0.6853 (3)	0.0114 (8)
C5	−0.0410 (6)	0.8603 (4)	0.7065 (3)	0.0142 (8)
H5	−0.1718	0.8743	0.7507	0.017*
C6	−0.0248 (7)	0.7721 (4)	0.6625 (3)	0.0144 (8)
H6	−0.1423	0.7239	0.6776	0.017*
C7	0.3204 (7)	0.9096 (4)	0.9434 (3)	0.0119 (8)
C8	0.7027 (7)	0.7845 (4)	0.9855 (4)	0.0179 (9)
H8	0.8417	0.7468	0.9596	0.022*
C9	0.7061 (7)	0.7567 (4)	1.1064 (4)	0.0185 (9)
H9	0.8443	0.7036	1.1607	0.022*
C10	0.5050 (7)	0.8081 (4)	1.1447 (4)	0.0152 (9)
H10	0.4981	0.7913	1.2267	0.018*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.01540 (15)	0.01568 (15)	0.01591 (15)	−0.00270 (10)	0.00046 (10)	−0.00950 (11)
I2	0.03577 (19)	0.02479 (18)	0.02029 (17)	−0.01282 (14)	0.00720 (12)	−0.01083 (14)
S1	0.0154 (5)	0.0133 (5)	0.0099 (5)	−0.0035 (4)	0.0020 (4)	−0.0073 (4)
O1	0.0222 (16)	0.0174 (16)	0.0158 (15)	−0.0127 (12)	0.0074 (11)	−0.0097 (13)
O2	0.0175 (15)	0.0151 (16)	0.0119 (15)	0.0021 (12)	−0.0028 (10)	−0.0067 (12)
N1	0.0110 (18)	0.0147 (19)	0.0125 (18)	0.0005 (14)	−0.0018 (13)	−0.0081 (15)
N2	0.0132 (17)	0.0140 (18)	0.0094 (16)	−0.0020 (13)	0.0005 (12)	−0.0074 (14)
N3	0.0132 (18)	0.0166 (19)	0.0118 (18)	−0.0009 (14)	0.0011 (12)	−0.0076 (15)
N4	0.0144 (18)	0.0147 (19)	0.0114 (17)	−0.0054 (14)	0.0027 (13)	−0.0089 (15)
C1	0.012 (2)	0.011 (2)	0.0091 (19)	0.0011 (15)	−0.0016 (14)	−0.0063 (16)
C2	0.014 (2)	0.017 (2)	0.013 (2)	−0.0025 (16)	0.0046 (15)	−0.0091 (17)

C3	0.011 (2)	0.017 (2)	0.012 (2)	−0.0057 (16)	0.0021 (14)	−0.0076 (17)
C4	0.011 (2)	0.012 (2)	0.0093 (19)	−0.0020 (15)	−0.0018 (14)	−0.0040 (16)
C5	0.010 (2)	0.022 (2)	0.012 (2)	−0.0035 (16)	0.0032 (14)	−0.0097 (18)
C6	0.011 (2)	0.017 (2)	0.016 (2)	−0.0061 (16)	0.0014 (15)	−0.0080 (18)
C7	0.015 (2)	0.010 (2)	0.012 (2)	−0.0041 (15)	0.0003 (14)	−0.0060 (16)
C8	0.016 (2)	0.018 (2)	0.019 (2)	−0.0030 (17)	0.0023 (16)	−0.0100 (19)
C9	0.017 (2)	0.019 (2)	0.017 (2)	−0.0013 (17)	−0.0020 (16)	−0.0078 (18)
C10	0.018 (2)	0.015 (2)	0.013 (2)	−0.0064 (17)	0.0000 (15)	−0.0065 (17)

Geometric parameters (Å, º)

I1—I2	3.3647 (4)	C1—C6	1.376 (5)
I2—I2^{i}	2.7623 (6)	C1—C2	1.384 (5)
S1—O1	1.443 (3)	C2—C3	1.373 (5)
S1—O2	1.455 (3)	C2—H2	0.9500
S1—N2	1.598 (3)	C3—C4	1.394 (5)
S1—C4	1.771 (4)	C3—H3	0.9500
N1—C1	1.460 (5)	C4—C5	1.382 (5)
N1—H1N	0.85 (4)	C5—C6	1.388 (5)
N1—H2N	0.84 (4)	C5—H5	0.9500
N1—H3N	0.96 (4)	C6—H6	0.9500
N2—C7	1.347 (5)	C8—C9	1.388 (5)
N3—C8	1.334 (5)	C8—H8	0.9500
N3—C7	1.350 (5)	C9—C10	1.364 (5)
N4—C10	1.353 (5)	C9—H9	0.9500
N4—C7	1.371 (5)	C10—H10	0.9500
N4—H4N	0.91 (4)		
I2^{i}—I2—I1	174.992 (16)	C2—C3—C4	119.5 (3)
O1—S1—O2	115.97 (17)	C2—C3—H3	120.3
O1—S1—N2	114.37 (16)	C4—C3—H3	120.3
O2—S1—N2	103.10 (16)	C5—C4—C3	121.0 (4)
O1—S1—C4	107.10 (17)	C5—C4—S1	120.1 (3)
O2—S1—C4	105.90 (16)	C3—C4—S1	118.7 (3)
N2—S1—C4	110.06 (17)	C4—C5—C6	119.5 (4)
C1—N1—H1N	107 (3)	C4—C5—H5	120.2
C1—N1—H2N	108 (3)	C6—C5—H5	120.2
H1N—N1—H2N	107 (4)	C1—C6—C5	118.7 (3)
C1—N1—H3N	110 (2)	C1—C6—H6	120.6
H1N—N1—H3N	118 (4)	C5—C6—H6	120.6
H2N—N1—H3N	106 (3)	N2—C7—N3	125.1 (4)
C7—N2—S1	120.8 (3)	N2—C7—N4	114.0 (3)
C8—N3—C7	116.5 (3)	N3—C7—N4	120.8 (3)
C10—N4—C7	122.3 (3)	N3—C8—C9	124.6 (4)
C10—N4—H4N	122 (2)	N3—C8—H8	117.7
C7—N4—H4N	116 (2)	C9—C8—H8	117.7
C6—C1—C2	122.2 (4)	C10—C9—C8	117.9 (4)
C6—C1—N1	119.6 (3)	C10—C9—H9	121.0

C2—C1—N1	118.1 (3)	C8—C9—H9	121.0
C3—C2—C1	119.0 (4)	N4—C10—C9	117.9 (4)
C3—C2—H2	120.5	N4—C10—H10	121.1
C1—C2—H2	120.5	C9—C10—H10	121.1
O1—S1—N2—C7	−50.9 (3)	S1—C4—C5—C6	174.9 (3)
O2—S1—N2—C7	−177.7 (3)	C2—C1—C6—C5	0.4 (6)
C4—S1—N2—C7	69.7 (3)	N1—C1—C6—C5	−176.9 (3)
C6—C1—C2—C3	1.4 (6)	C4—C5—C6—C1	−1.2 (6)
N1—C1—C2—C3	178.7 (4)	S1—N2—C7—N3	−12.7 (5)
C1—C2—C3—C4	−2.4 (6)	S1—N2—C7—N4	167.5 (3)
C2—C3—C4—C5	1.6 (6)	C8—N3—C7—N2	−178.6 (4)
C2—C3—C4—S1	−173.2 (3)	C8—N3—C7—N4	1.2 (5)
O1—S1—C4—C5	−169.1 (3)	C10—N4—C7—N2	179.5 (3)
O2—S1—C4—C5	−44.7 (3)	C10—N4—C7—N3	−0.3 (5)
N2—S1—C4—C5	66.0 (3)	C7—N3—C8—C9	−1.9 (6)
O1—S1—C4—C3	5.8 (4)	N3—C8—C9—C10	1.6 (6)
O2—S1—C4—C3	130.1 (3)	C7—N4—C10—C9	−0.1 (5)
N2—S1—C4—C3	−119.1 (3)	C8—C9—C10—N4	−0.5 (6)
C3—C4—C5—C6	0.2 (6)		

Symmetry code: (i) −*x*−1, −*y*+1, −*z*+2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···I1^{ii}	0.85 (4)	2.64 (4)	3.482 (4)	172 (4)
N1—H2*N*···O2^{iii}	0.84 (4)	2.00 (4)	2.798 (4)	158 (4)
N1—H3*N*···I1	0.96 (4)	2.59 (4)	3.524 (3)	165 (3)
N4—H4*N*···N2^{iv}	0.91 (4)	2.00 (4)	2.906 (5)	174 (4)

Symmetry codes: (ii) *x*+1, *y*, *z*; (iii) −*x*, −*y*+2, −*z*+1; (iv) −*x*, −*y*+2, −*z*+2.

(V) 2-{[(4-Azaniumylphenyl)sulfonyl]azanidyl}pyrimidin-1-ium tetrafluoroborate monohydrate

Crystal data

$C_{10}H_{11}N_4O_2S^+·BF_4^-·H_2O$
M_r = 356.11
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 5.8539 (1) Å
b = 11.3629 (3) Å
c = 21.2080 (6) Å
β = 90.982 (2)°
V = 1410.49 (6) Å^3
Z = 4
$F(000)$ = 728
D_x = 1.677 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4378 reflections
θ = 3.4–29.8°
μ = 0.30 mm^{-1}
T = 123 K
Fragment, light yellow
0.20 × 0.12 × 0.05 mm

Data collection

Oxford Diffraction Xcalibur E diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.874, T_{max} = 1.000

8710 measured reflections
3549 independent reflections
2955 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 29.0°$, $\theta_{min} = 3.4°$
$h = -7 \rightarrow 7$
$k = -15 \rightarrow 11$
$l = -24 \rightarrow 27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.089$
$S = 1.03$
3549 reflections
232 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0362P)^2 + 0.6973P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlisPro, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.21736 (6)	0.24493 (3)	1.039691 (17)	0.01188 (10)
F1	−0.15946 (17)	0.20817 (10)	0.74824 (5)	0.0301 (3)
F2	0.09745 (19)	0.18041 (11)	0.82770 (5)	0.0312 (3)
F3	0.2036 (2)	0.15335 (12)	0.72644 (6)	0.0434 (3)
F4	0.13039 (17)	0.33817 (10)	0.76257 (5)	0.0273 (3)
O1	0.44811 (18)	0.27762 (10)	1.05680 (5)	0.0175 (2)
O2	0.05509 (19)	0.25011 (10)	1.09069 (5)	0.0164 (2)
O1W	−0.4027 (2)	0.41744 (12)	0.76066 (6)	0.0245 (3)
N1	−0.0902 (2)	0.55457 (13)	0.82772 (7)	0.0143 (3)
N2	0.1822 (2)	0.11597 (11)	1.01075 (6)	0.0120 (3)
N3	0.2260 (2)	−0.01928 (12)	0.93253 (6)	0.0126 (3)
N4	0.4790 (2)	0.14123 (12)	0.93657 (6)	0.0156 (3)
C1	−0.0140 (2)	0.47962 (14)	0.88004 (7)	0.0117 (3)
C2	0.2045 (3)	0.49308 (14)	0.90438 (8)	0.0157 (3)
H2	0.3052	0.5501	0.8875	0.019*
C3	0.2735 (2)	0.42144 (14)	0.95408 (7)	0.0145 (3)
H3	0.4245	0.4276	0.9709	0.017*

C4	0.1225 (2)	0.34096 (13)	0.97914 (7)	0.0115 (3)
C5	−0.0992 (2)	0.33031 (14)	0.95543 (7)	0.0143 (3)
H5	−0.2030	0.2765	0.9737	0.017*
C6	−0.1662 (2)	0.39939 (14)	0.90476 (7)	0.0143 (3)
H6	−0.3157	0.3919	0.8870	0.017*
C7	0.2996 (2)	0.08243 (14)	0.95992 (7)	0.0114 (3)
C8	0.3297 (3)	−0.06628 (15)	0.88228 (7)	0.0153 (3)
H8	0.2755	−0.1376	0.8641	0.018*
C9	0.5143 (3)	−0.00977 (15)	0.85782 (8)	0.0177 (3)
H9	0.5934	−0.0405	0.8227	0.021*
C10	0.5806 (3)	0.09446 (16)	0.88665 (8)	0.0184 (3)
H10	0.7071	0.1354	0.8697	0.022*
B1	0.0703 (3)	0.21971 (18)	0.76723 (9)	0.0183 (4)
H1N	0.030 (4)	0.5713 (19)	0.8011 (10)	0.029 (6)*
H2N	−0.133 (3)	0.623 (2)	0.8436 (9)	0.025 (5)*
H3N	−0.206 (4)	0.520 (2)	0.8040 (11)	0.040 (6)*
H4N	0.103 (3)	−0.0515 (17)	0.9493 (9)	0.023 (5)*
H1W	−0.5492 (16)	0.407 (2)	0.7599 (12)	0.060 (8)*
H2W	−0.355 (4)	0.3449 (11)	0.7561 (12)	0.053 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01499 (17)	0.00899 (19)	0.01162 (19)	−0.00181 (13)	−0.00057 (13)	−0.00011 (15)
F1	0.0210 (5)	0.0234 (6)	0.0456 (7)	0.0003 (4)	−0.0106 (5)	−0.0098 (5)
F2	0.0366 (6)	0.0335 (7)	0.0236 (6)	0.0045 (5)	0.0003 (4)	0.0068 (5)
F3	0.0405 (7)	0.0512 (8)	0.0388 (7)	0.0177 (6)	0.0144 (5)	−0.0141 (6)
F4	0.0243 (5)	0.0253 (6)	0.0322 (6)	−0.0070 (4)	−0.0003 (4)	0.0046 (5)
O1	0.0184 (5)	0.0145 (6)	0.0194 (6)	−0.0038 (4)	−0.0066 (4)	0.0021 (5)
O2	0.0242 (6)	0.0122 (6)	0.0131 (5)	−0.0013 (4)	0.0037 (4)	−0.0010 (5)
O1W	0.0182 (6)	0.0221 (7)	0.0331 (7)	−0.0004 (5)	−0.0037 (5)	−0.0056 (6)
N1	0.0184 (6)	0.0114 (7)	0.0132 (7)	0.0001 (5)	0.0005 (5)	0.0014 (6)
N2	0.0154 (6)	0.0079 (6)	0.0128 (6)	−0.0026 (5)	0.0016 (5)	−0.0002 (5)
N3	0.0143 (6)	0.0100 (6)	0.0135 (6)	−0.0008 (5)	0.0010 (5)	0.0010 (5)
N4	0.0149 (6)	0.0153 (7)	0.0166 (7)	−0.0036 (5)	0.0026 (5)	0.0006 (6)
C1	0.0165 (7)	0.0098 (7)	0.0087 (7)	0.0011 (6)	0.0011 (5)	0.0001 (6)
C2	0.0166 (7)	0.0122 (8)	0.0182 (8)	−0.0046 (6)	0.0018 (6)	0.0010 (7)
C3	0.0131 (6)	0.0134 (8)	0.0169 (8)	−0.0031 (6)	−0.0013 (5)	−0.0006 (7)
C4	0.0146 (7)	0.0090 (7)	0.0110 (7)	−0.0002 (5)	0.0009 (5)	−0.0012 (6)
C5	0.0136 (7)	0.0133 (8)	0.0160 (7)	−0.0033 (6)	0.0020 (5)	0.0010 (7)
C6	0.0123 (6)	0.0155 (8)	0.0152 (7)	−0.0009 (6)	0.0002 (5)	0.0003 (7)
C7	0.0122 (6)	0.0097 (7)	0.0122 (7)	0.0005 (5)	−0.0023 (5)	0.0006 (6)
C8	0.0202 (7)	0.0123 (8)	0.0135 (7)	0.0025 (6)	0.0001 (6)	−0.0007 (7)
C9	0.0198 (7)	0.0193 (9)	0.0142 (8)	0.0023 (6)	0.0037 (6)	−0.0017 (7)
C10	0.0157 (7)	0.0218 (9)	0.0179 (8)	−0.0019 (6)	0.0032 (6)	0.0020 (7)
B1	0.0155 (8)	0.0201 (10)	0.0194 (9)	0.0029 (7)	0.0023 (6)	−0.0027 (8)

Geometric parameters (Å, º)

S1—O1	1.4413 (11)	N4—C10	1.334 (2)
S1—O2	1.4528 (11)	N4—C7	1.3463 (19)
S1—N2	1.6006 (13)	C1—C2	1.379 (2)
S1—C4	1.7675 (16)	C1—C6	1.384 (2)
F1—B1	1.404 (2)	C2—C3	1.386 (2)
F2—B1	1.365 (2)	C2—H2	0.9500
F3—B1	1.396 (2)	C3—C4	1.384 (2)
F4—B1	1.395 (2)	C3—H3	0.9500
O1W—H1W	0.867 (10)	C4—C5	1.389 (2)
O1W—H2W	0.877 (9)	C5—C6	1.382 (2)
N1—C1	1.463 (2)	C5—H5	0.9500
N1—H1N	0.93 (2)	C6—H6	0.9500
N1—H2N	0.88 (2)	C8—C9	1.367 (2)
N1—H3N	0.92 (2)	C8—H8	0.9500
N2—C7	1.3433 (18)	C9—C10	1.385 (2)
N3—C8	1.3460 (19)	C9—H9	0.9500
N3—C7	1.360 (2)	C10—H10	0.9500
N3—H4N	0.89 (2)		
O1—S1—O2	115.10 (7)	C3—C4—C5	121.16 (14)
O1—S1—N2	116.50 (7)	C3—C4—S1	119.46 (11)
O2—S1—N2	103.96 (7)	C5—C4—S1	119.30 (12)
O1—S1—C4	107.78 (7)	C6—C5—C4	118.88 (14)
O2—S1—C4	108.33 (7)	C6—C5—H5	120.6
N2—S1—C4	104.47 (7)	C4—C5—H5	120.6
H1W—O1W—H2W	100.6 (17)	C5—C6—C1	119.49 (14)
C1—N1—H1N	110.8 (13)	C5—C6—H6	120.3
C1—N1—H2N	107.8 (13)	C1—C6—H6	120.3
H1N—N1—H2N	106.0 (18)	N2—C7—N4	124.56 (14)
C1—N1—H3N	112.4 (15)	N2—C7—N3	114.91 (13)
H1N—N1—H3N	108.2 (19)	N4—C7—N3	120.52 (13)
H2N—N1—H3N	111.5 (19)	N3—C8—C9	118.98 (15)
C7—N2—S1	120.22 (11)	N3—C8—H8	120.5
C8—N3—C7	122.10 (13)	C9—C8—H8	120.5
C8—N3—H4N	122.3 (13)	C8—C9—C10	116.84 (15)
C7—N3—H4N	115.6 (13)	C8—C9—H9	121.6
C10—N4—C7	117.11 (14)	C10—C9—H9	121.6
C2—C1—C6	122.02 (14)	N4—C10—C9	124.44 (15)
C2—C1—N1	119.22 (14)	N4—C10—H10	117.8
C6—C1—N1	118.73 (13)	C9—C10—H10	117.8
C1—C2—C3	118.40 (14)	F2—B1—F4	110.92 (15)
C1—C2—H2	120.8	F2—B1—F3	110.39 (15)
C3—C2—H2	120.8	F4—B1—F3	109.46 (15)
C4—C3—C2	120.00 (14)	F2—B1—F1	109.54 (14)
C4—C3—H3	120.0	F4—B1—F1	108.13 (14)
C2—C3—H3	120.0	F3—B1—F1	108.33 (15)

O1—S1—N2—C7	−55.37 (14)	S1—C4—C5—C6	−174.93 (12)
O2—S1—N2—C7	176.88 (12)	C4—C5—C6—C1	−1.8 (2)
C4—S1—N2—C7	63.38 (13)	C2—C1—C6—C5	0.1 (2)
C6—C1—C2—C3	1.7 (2)	N1—C1—C6—C5	−178.09 (14)
N1—C1—C2—C3	179.84 (14)	S1—N2—C7—N4	12.5 (2)
C1—C2—C3—C4	−1.7 (2)	S1—N2—C7—N3	−168.26 (11)
C2—C3—C4—C5	−0.1 (2)	C10—N4—C7—N2	178.32 (15)
C2—C3—C4—S1	176.70 (12)	C10—N4—C7—N3	−0.9 (2)
O1—S1—C4—C3	5.16 (15)	C8—N3—C7—N2	−178.12 (13)
O2—S1—C4—C3	130.29 (13)	C8—N3—C7—N4	1.2 (2)
N2—S1—C4—C3	−119.36 (13)	C7—N3—C8—C9	−0.3 (2)
O1—S1—C4—C5	−178.00 (12)	N3—C8—C9—C10	−0.8 (2)
O2—S1—C4—C5	−52.87 (14)	C7—N4—C10—C9	−0.2 (2)
N2—S1—C4—C5	57.48 (14)	C8—C9—C10—N4	1.1 (3)
C3—C4—C5—C6	1.9 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···F3^{i}	0.93 (2)	1.92 (2)	2.7912 (17)	156.0 (19)
N1—H2*N*···O2^{ii}	0.88 (2)	2.05 (2)	2.8196 (19)	144.7 (18)
N1—H3*N*···O1*W*	0.92 (2)	1.87 (3)	2.7762 (19)	166 (2)
N3—H4*N*···N2^{iii}	0.89 (2)	2.02 (2)	2.9092 (17)	176.8 (19)
N3—H4*N*···O2^{iii}	0.89 (2)	2.58 (2)	3.1307 (17)	121.2 (15)
O1*W*—H1*W*···F4^{iv}	0.87 (1)	2.03 (1)	2.8790 (15)	165 (2)
O1*W*—H2*W*···F1	0.88 (1)	1.94 (1)	2.7863 (17)	163 (2)

Symmetry codes: (i) −*x*+1/2, *y*+1/2, −*z*+3/2; (ii) −*x*, −*y*+1, −*z*+2; (iii) −*x*, −*y*, −*z*+2; (iv) *x*−1, *y*, *z*.

(VI) 2-{[(4-Azaniumylphenyl)sulfonyl]azanidyl}pyrimidin-1-ium nitrate

Crystal data

$C_{10}H_{11}N_4O_2S^+\cdot NO_3^-$
M_r = 313.30
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 5.4705 (4) Å
b = 10.1235 (7) Å
c = 11.9956 (8) Å
α = 86.708 (6)°
β = 85.489 (6)°
γ = 74.394 (7)°
V = 637.40 (8) Å^3
Z = 2
F(000) = 324
D_x = 1.632 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3307 reflections
θ = 3.4–29.4°
μ = 0.29 mm^{-1}
T = 123 K
Fragment, light yellow
0.26 × 0.15 × 0.06 mm

Data collection

Oxford Diffraction Xcalibur E diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.897, T_{max} = 1.000
7390 measured reflections
2977 independent reflections
2429 reflections with $I > 2\sigma(I)$

R_{int} = 0.026
θ_{max} = 28.0°, θ_{min} = 3.4°
h = −6→7
k = −13→13
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.110
S = 1.04
2977 reflections
206 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0489P)^2 + 0.4298P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlisPro, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
S1	0.31611 (10)	−0.07161 (5)	0.77390 (4)	0.01458 (14)
O1	0.5490 (3)	−0.18213 (14)	0.76593 (12)	0.0182 (3)
O2	0.0876 (3)	−0.10972 (16)	0.75678 (12)	0.0215 (3)
O3	0.0278 (3)	0.5197 (2)	0.26381 (18)	0.0504 (6)
O4	−0.3521 (3)	0.50354 (18)	0.24688 (14)	0.0311 (4)
O5	−0.1819 (3)	0.45362 (15)	0.40662 (12)	0.0207 (3)
N1	0.3888 (4)	0.35138 (19)	0.41482 (15)	0.0169 (4)
N2	0.3246 (3)	−0.00844 (18)	0.89180 (14)	0.0159 (4)
N3	0.2015 (3)	0.16323 (19)	1.01576 (14)	0.0166 (4)
N4	−0.0507 (3)	0.17178 (19)	0.86457 (14)	0.0187 (4)
N5	−0.1690 (4)	0.49396 (19)	0.30339 (16)	0.0222 (4)
C1	0.3766 (4)	0.2491 (2)	0.50395 (16)	0.0147 (4)
C2	0.2027 (4)	0.1732 (2)	0.49778 (17)	0.0159 (4)
H2	0.0979	0.1869	0.4364	0.019*
C3	0.1839 (4)	0.0768 (2)	0.58262 (16)	0.0154 (4)
H3	0.0623	0.0255	0.5810	0.018*
C4	0.3440 (4)	0.0559 (2)	0.66987 (16)	0.0132 (4)
C5	0.5219 (4)	0.1303 (2)	0.67457 (17)	0.0158 (4)

H5	0.6323	0.1139	0.7341	0.019*
C6	0.5361 (4)	0.2292 (2)	0.59076 (17)	0.0160 (4)
H6	0.6542	0.2825	0.5931	0.019*
C7	0.1540 (4)	0.1084 (2)	0.92134 (16)	0.0154 (4)
C8	0.0485 (4)	0.2791 (2)	1.05659 (17)	0.0192 (4)
H8	0.0846	0.3139	1.1235	0.023*
C9	−0.1597 (4)	0.3466 (2)	1.00104 (19)	0.0227 (5)
H9	−0.2724	0.4293	1.0273	0.027*
C10	−0.1989 (4)	0.2886 (2)	0.90410 (19)	0.0229 (5)
H10	−0.3410	0.3354	0.8634	0.027*
H1N	0.528 (6)	0.392 (3)	0.419 (2)	0.041 (8)*
H2N	0.253 (6)	0.417 (3)	0.415 (2)	0.041 (9)*
H3N	0.408 (5)	0.307 (3)	0.346 (2)	0.035 (8)*
H4N	0.336 (5)	0.119 (3)	1.046 (2)	0.026 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0165 (3)	0.0174 (3)	0.0102 (2)	−0.00517 (19)	−0.00247 (18)	0.00136 (18)
O1	0.0222 (8)	0.0169 (7)	0.0139 (7)	−0.0022 (6)	−0.0027 (6)	0.0002 (6)
O2	0.0239 (9)	0.0264 (8)	0.0180 (8)	−0.0135 (7)	−0.0056 (6)	0.0061 (6)
O3	0.0225 (10)	0.0633 (14)	0.0593 (14)	−0.0107 (9)	0.0046 (9)	0.0348 (11)
O4	0.0343 (10)	0.0356 (10)	0.0215 (9)	−0.0050 (8)	−0.0109 (7)	0.0052 (7)
O5	0.0255 (9)	0.0217 (8)	0.0147 (7)	−0.0060 (6)	−0.0035 (6)	0.0020 (6)
N1	0.0210 (10)	0.0149 (9)	0.0139 (9)	−0.0034 (8)	−0.0004 (7)	0.0008 (7)
N2	0.0170 (9)	0.0200 (9)	0.0093 (8)	−0.0020 (7)	−0.0033 (6)	0.0007 (7)
N3	0.0158 (9)	0.0205 (9)	0.0117 (8)	−0.0018 (7)	−0.0023 (7)	0.0026 (7)
N4	0.0166 (9)	0.0235 (9)	0.0136 (8)	−0.0012 (7)	−0.0032 (7)	0.0025 (7)
N5	0.0188 (10)	0.0213 (9)	0.0223 (10)	0.0003 (8)	0.0016 (8)	0.0040 (8)
C1	0.0160 (10)	0.0138 (9)	0.0114 (9)	0.0001 (8)	0.0019 (8)	0.0003 (7)
C2	0.0163 (11)	0.0191 (10)	0.0122 (9)	−0.0044 (8)	−0.0033 (8)	0.0017 (8)
C3	0.0135 (10)	0.0200 (10)	0.0138 (10)	−0.0067 (8)	−0.0007 (8)	0.0001 (8)
C4	0.0121 (10)	0.0145 (9)	0.0115 (9)	−0.0012 (7)	−0.0002 (7)	0.0002 (7)
C5	0.0138 (10)	0.0194 (10)	0.0140 (10)	−0.0031 (8)	−0.0042 (8)	−0.0015 (8)
C6	0.0146 (10)	0.0166 (10)	0.0176 (10)	−0.0050 (8)	−0.0004 (8)	−0.0021 (8)
C7	0.0154 (10)	0.0205 (10)	0.0103 (9)	−0.0061 (8)	0.0007 (8)	0.0026 (8)
C8	0.0240 (12)	0.0193 (10)	0.0135 (10)	−0.0050 (9)	0.0010 (8)	0.0001 (8)
C9	0.0231 (12)	0.0202 (11)	0.0205 (11)	0.0011 (9)	−0.0001 (9)	0.0004 (9)
C10	0.0192 (11)	0.0254 (11)	0.0204 (11)	−0.0005 (9)	−0.0025 (9)	0.0047 (9)

Geometric parameters (Å, º)

S1—O2	1.4365 (15)	N4—C7	1.346 (3)
S1—O1	1.4527 (15)	C1—C2	1.382 (3)
S1—N2	1.5941 (17)	C1—C6	1.382 (3)
S1—C4	1.771 (2)	C2—C3	1.386 (3)
O3—N5	1.228 (3)	C2—H2	0.9500
O4—N5	1.233 (2)	C3—C4	1.388 (3)

O5—N5	1.284 (2)	C3—H3	0.9500
N1—C1	1.455 (3)	C4—C5	1.388 (3)
N1—H1N	0.96 (3)	C5—C6	1.390 (3)
N1—H2N	0.85 (3)	C5—H5	0.9500
N1—H3N	0.95 (3)	C6—H6	0.9500
N2—C7	1.341 (3)	C8—C9	1.363 (3)
N3—C8	1.338 (3)	C8—H8	0.9500
N3—C7	1.362 (3)	C9—C10	1.389 (3)
N3—H4N	0.85 (3)	C9—H9	0.9500
N4—C10	1.330 (3)	C10—H10	0.9500

O2—S1—O1	115.35 (9)	C3—C2—H2	120.5
O2—S1—N2	115.72 (9)	C2—C3—C4	119.55 (19)
O1—S1—N2	103.48 (9)	C2—C3—H3	120.2
O2—S1—C4	107.13 (9)	C4—C3—H3	120.2
O1—S1—C4	107.81 (9)	C3—C4—C5	121.34 (19)
N2—S1—C4	106.87 (9)	C3—C4—S1	117.43 (15)
C1—N1—H1N	113.7 (17)	C5—C4—S1	121.22 (15)
C1—N1—H2N	112 (2)	C4—C5—C6	118.94 (18)
H1N—N1—H2N	107 (3)	C4—C5—H5	120.5
C1—N1—H3N	107.0 (17)	C6—C5—H5	120.5
H1N—N1—H3N	109 (2)	C1—C6—C5	119.34 (19)
H2N—N1—H3N	109 (2)	C1—C6—H6	120.3
C7—N2—S1	120.20 (14)	C5—C6—H6	120.3
C8—N3—C7	122.28 (19)	N2—C7—N4	124.84 (19)
C8—N3—H4N	122.6 (17)	N2—C7—N3	114.98 (18)
C7—N3—H4N	115.1 (17)	N4—C7—N3	120.18 (19)
C10—N4—C7	117.29 (19)	N3—C8—C9	119.2 (2)
O3—N5—O4	122.2 (2)	N3—C8—H8	120.4
O3—N5—O5	118.7 (2)	C9—C8—H8	120.4
O4—N5—O5	119.15 (19)	C8—C9—C10	116.7 (2)
C2—C1—C6	121.87 (19)	C8—C9—H9	121.7
C2—C1—N1	117.73 (18)	C10—C9—H9	121.7
C6—C1—N1	120.40 (19)	N4—C10—C9	124.3 (2)
C1—C2—C3	118.92 (18)	N4—C10—H10	117.8
C1—C2—H2	120.5	C9—C10—H10	117.8

O2—S1—N2—C7	−61.49 (19)	S1—C4—C5—C6	179.99 (15)
O1—S1—N2—C7	171.35 (16)	C2—C1—C6—C5	−0.1 (3)
C4—S1—N2—C7	57.69 (18)	N1—C1—C6—C5	−179.59 (18)
C6—C1—C2—C3	1.6 (3)	C4—C5—C6—C1	−1.2 (3)
N1—C1—C2—C3	−178.85 (18)	S1—N2—C7—N4	8.6 (3)
C1—C2—C3—C4	−1.8 (3)	S1—N2—C7—N3	−170.79 (14)
C2—C3—C4—C5	0.5 (3)	C10—N4—C7—N2	−178.8 (2)
C2—C3—C4—S1	−178.48 (15)	C10—N4—C7—N3	0.6 (3)
O2—S1—C4—C3	−11.74 (19)	C8—N3—C7—N2	−179.64 (19)
O1—S1—C4—C3	112.98 (16)	C8—N3—C7—N4	1.0 (3)
N2—S1—C4—C3	−136.34 (16)	C7—N3—C8—C9	−1.3 (3)

O2—S1—C4—C5	169.27 (16)	N3—C8—C9—C10	0.2 (3)
O1—S1—C4—C5	−66.01 (18)	C7—N4—C10—C9	−1.8 (3)
N2—S1—C4—C5	44.67 (19)	C8—C9—C10—N4	1.4 (4)
C3—C4—C5—C6	1.0 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O5^{i}	0.96 (3)	1.85 (3)	2.803 (3)	171 (3)
N1—H1*N*···O4^{i}	0.96 (3)	2.42 (3)	2.968 (3)	116 (2)
N1—H2*N*···O3	0.85 (3)	2.31 (3)	2.912 (3)	128 (3)
N1—H2*N*···O5	0.85 (3)	2.32 (3)	3.021 (3)	140 (3)
N1—H3*N*···O1^{ii}	0.95 (3)	1.85 (3)	2.784 (2)	166 (3)
N3—H4*N*···N2^{iii}	0.85 (3)	2.06 (3)	2.911 (3)	176 (2)
N3—H4*N*···O1^{iii}	0.85 (3)	2.55 (3)	3.085 (2)	122 (2)

Symmetry codes: (i) x+1, y, z; (ii) −x+1, −y, −z+1; (iii) −x+1, −y, −z+2.

(VII) 4-[(Pyrimidin-2-yl)sulfamoyl]anilinium ethanesulfonate

Crystal data

$C_{10}H_{11}N_4O_2S^+·C_2H_5O_3S^-$
M_r = 360.41
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 5.5269 (4) Å
b = 34.979 (3) Å
c = 8.4395 (6) Å
β = 93.102 (7)°
V = 1629.2 (2) Å^3
Z = 4
$F(000)$ = 752
D_x = 1.469 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2991 reflections
θ = 3.4–28.7°
μ = 0.36 mm^{-1}
T = 123 K
Needle, colourless
0.4 × 0.03 × 0.02 mm

Data collection

Oxford Diffraction Xcalibur E diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.908, T_{max} = 1.000
19991 measured reflections
3515 independent reflections
2212 reflections with $I > 2\sigma(I)$
R_{int} = 0.104
θ_{max} = 27.0°, θ_{min} = 3.4°
h = −7→7
k = −43→44
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.112
S = 1.01
3515 reflections
222 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 0.6311P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlisPro, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.28192 (13)	0.44093 (2)	0.77910 (9)	0.0177 (2)
S2	0.00973 (13)	0.27930 (2)	0.23275 (9)	0.0181 (2)
O1	0.4676 (4)	0.45420 (6)	0.8902 (2)	0.0224 (5)
O2	0.0371 (4)	0.44002 (6)	0.8241 (2)	0.0213 (5)
O3	−0.1498 (4)	0.24673 (6)	0.2104 (3)	0.0282 (6)
O4	−0.0356 (4)	0.30107 (7)	0.3739 (3)	0.0271 (6)
O5	0.2670 (3)	0.26823 (6)	0.2301 (3)	0.0222 (5)
N1	0.4972 (5)	0.28671 (8)	0.5241 (3)	0.0215 (6)
H1N	0.452 (6)	0.2671 (7)	0.586 (3)	0.032*
H2N	0.656 (2)	0.2841 (10)	0.505 (4)	0.032*
H3N	0.420 (5)	0.2818 (9)	0.428 (2)	0.032*
N2	0.3082 (5)	0.46787 (8)	0.6229 (3)	0.0184 (6)
N3	0.0015 (5)	0.43804 (8)	0.4667 (3)	0.0224 (6)
N4	0.2795 (4)	0.48159 (7)	0.3562 (3)	0.0184 (6)
C1	0.4512 (5)	0.32414 (9)	0.5930 (4)	0.0166 (7)
C2	0.2449 (5)	0.32892 (9)	0.6757 (4)	0.0189 (7)
H2	0.1388	0.3081	0.6919	0.023*
C3	0.1965 (5)	0.36487 (9)	0.7345 (4)	0.0180 (7)
H3	0.0538	0.3690	0.7901	0.022*
C4	0.3537 (5)	0.39464 (9)	0.7130 (4)	0.0155 (7)
C5	0.5653 (5)	0.38924 (9)	0.6337 (4)	0.0186 (7)
H5	0.6750	0.4098	0.6217	0.022*
C6	0.6134 (5)	0.35368 (9)	0.5727 (4)	0.0183 (7)
H6	0.7563	0.3495	0.5175	0.022*
C7	0.1892 (5)	0.46205 (9)	0.4759 (4)	0.0167 (7)
C8	−0.1070 (6)	0.43437 (10)	0.3223 (4)	0.0261 (8)
H8	−0.2436	0.4179	0.3103	0.031*
C9	−0.0331 (6)	0.45291 (9)	0.1902 (4)	0.0232 (8)
H9	−0.1136	0.4497	0.0887	0.028*
C10	0.1660 (6)	0.47650 (9)	0.2140 (4)	0.0218 (8)
H10	0.2247	0.4897	0.1254	0.026*
C11	−0.0402 (6)	0.30986 (10)	0.0676 (4)	0.0294 (9)
H11A	0.0793	0.3310	0.0752	0.035*

H11B	−0.0128	0.2953	−0.0305	0.035*
C12	−0.2942 (6)	0.32674 (12)	0.0553 (5)	0.0443 (11)
H12A	−0.4136	0.3060	0.0450	0.066*
H12B	−0.3112	0.3434	−0.0380	0.066*
H12C	−0.3215	0.3417	0.1510	0.066*
H4N	0.442 (6)	0.4822 (10)	0.627 (4)	0.031 (10)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0194 (4)	0.0175 (4)	0.0167 (4)	−0.0018 (3)	0.0050 (3)	−0.0007 (4)
S2	0.0156 (4)	0.0213 (5)	0.0172 (4)	−0.0003 (3)	0.0003 (3)	−0.0022 (4)
O1	0.0268 (12)	0.0252 (13)	0.0152 (12)	−0.0049 (10)	0.0015 (9)	−0.0029 (10)
O2	0.0216 (11)	0.0223 (13)	0.0208 (13)	0.0024 (10)	0.0084 (9)	−0.0009 (10)
O3	0.0204 (12)	0.0280 (14)	0.0354 (15)	−0.0057 (10)	−0.0078 (10)	0.0004 (12)
O4	0.0238 (12)	0.0355 (15)	0.0220 (13)	0.0042 (11)	0.0002 (10)	−0.0100 (11)
O5	0.0139 (11)	0.0300 (14)	0.0228 (13)	0.0032 (10)	0.0025 (9)	−0.0052 (10)
N1	0.0216 (15)	0.0196 (16)	0.0230 (17)	0.0020 (13)	−0.0036 (13)	−0.0039 (13)
N2	0.0195 (14)	0.0184 (16)	0.0176 (15)	−0.0054 (13)	0.0032 (12)	0.0020 (12)
N3	0.0243 (15)	0.0229 (16)	0.0202 (16)	−0.0062 (13)	0.0036 (12)	0.0001 (13)
N4	0.0216 (14)	0.0168 (15)	0.0173 (15)	0.0011 (11)	0.0064 (11)	0.0017 (12)
C1	0.0151 (16)	0.0192 (18)	0.0150 (17)	0.0037 (13)	−0.0051 (13)	−0.0029 (14)
C2	0.0184 (17)	0.0160 (18)	0.0219 (18)	−0.0018 (14)	−0.0027 (14)	0.0019 (15)
C3	0.0163 (16)	0.0204 (18)	0.0179 (18)	−0.0006 (14)	0.0049 (13)	0.0028 (15)
C4	0.0164 (16)	0.0158 (17)	0.0141 (17)	−0.0006 (13)	−0.0009 (13)	0.0011 (14)
C5	0.0172 (16)	0.0197 (18)	0.0189 (18)	−0.0050 (14)	0.0017 (13)	0.0012 (15)
C6	0.0135 (16)	0.0231 (19)	0.0186 (18)	0.0003 (14)	0.0040 (13)	−0.0029 (15)
C7	0.0181 (16)	0.0156 (18)	0.0168 (18)	0.0037 (13)	0.0055 (13)	−0.0021 (14)
C8	0.0251 (18)	0.027 (2)	0.026 (2)	−0.0061 (15)	0.0014 (15)	−0.0049 (16)
C9	0.0280 (18)	0.026 (2)	0.0154 (18)	−0.0035 (15)	0.0004 (14)	−0.0002 (15)
C10	0.0316 (19)	0.0203 (19)	0.0141 (18)	0.0043 (15)	0.0071 (14)	0.0009 (15)
C11	0.0266 (19)	0.035 (2)	0.026 (2)	−0.0025 (16)	0.0045 (15)	0.0090 (17)
C12	0.034 (2)	0.054 (3)	0.045 (3)	0.0042 (19)	−0.0019 (19)	0.021 (2)

Geometric parameters (Å, º)

S1—O2	1.425 (2)	C2—C3	1.383 (4)
S1—O1	1.430 (2)	C2—H2	0.9500
S1—N2	1.633 (3)	C3—C4	1.375 (4)
S1—C4	1.765 (3)	C3—H3	0.9500
S2—O3	1.447 (2)	C4—C5	1.391 (4)
S2—O4	1.447 (2)	C5—C6	1.377 (4)
S2—O5	1.475 (2)	C5—H5	0.9500
S2—C11	1.766 (3)	C6—H6	0.9500
N1—C1	1.460 (4)	C8—C9	1.371 (4)
N1—H1N	0.905 (10)	C8—H8	0.9500
N1—H2N	0.902 (10)	C9—C10	1.381 (4)
N1—H3N	0.913 (10)	C9—H9	0.9500

N2—C7	1.388 (4)	C10—H10	0.9500
N2—H4N	0.89 (3)	C11—C12	1.521 (5)
N3—C7	1.334 (4)	C11—H11A	0.9900
N3—C8	1.335 (4)	C11—H11B	0.9900
N4—C10	1.336 (4)	C12—H12A	0.9800
N4—C7	1.338 (4)	C12—H12B	0.9800
C1—C2	1.379 (4)	C12—H12C	0.9800
C1—C6	1.385 (4)		

O2—S1—O1	119.39 (13)	C3—C4—S1	119.9 (2)
O2—S1—N2	110.80 (13)	C5—C4—S1	119.1 (2)
O1—S1—N2	104.48 (14)	C6—C5—C4	119.1 (3)
O2—S1—C4	107.25 (14)	C6—C5—H5	120.4
O1—S1—C4	109.85 (13)	C4—C5—H5	120.4
N2—S1—C4	104.06 (14)	C5—C6—C1	119.2 (3)
O3—S2—O4	112.99 (14)	C5—C6—H6	120.4
O3—S2—O5	111.87 (13)	C1—C6—H6	120.4
O4—S2—O5	111.13 (13)	N3—C7—N4	126.8 (3)
O3—S2—C11	107.77 (15)	N3—C7—N2	118.3 (3)
O4—S2—C11	107.73 (16)	N4—C7—N2	115.0 (3)
O5—S2—C11	104.87 (14)	N3—C8—C9	123.9 (3)
C1—N1—H1N	113 (2)	N3—C8—H8	118.0
C1—N1—H2N	111 (2)	C9—C8—H8	118.0
H1N—N1—H2N	109 (3)	C8—C9—C10	115.8 (3)
C1—N1—H3N	116 (2)	C8—C9—H9	122.1
H1N—N1—H3N	104 (3)	C10—C9—H9	122.1
H2N—N1—H3N	104 (3)	N4—C10—C9	122.8 (3)
C7—N2—S1	125.4 (2)	N4—C10—H10	118.6
C7—N2—H4N	118 (2)	C9—C10—H10	118.6
S1—N2—H4N	113 (2)	C12—C11—S2	113.1 (2)
C7—N3—C8	115.0 (3)	C12—C11—H11A	109.0
C10—N4—C7	115.7 (3)	S2—C11—H11A	109.0
C2—C1—C6	122.1 (3)	C12—C11—H11B	109.0
C2—C1—N1	118.5 (3)	S2—C11—H11B	109.0
C6—C1—N1	119.5 (3)	H11A—C11—H11B	107.8
C1—C2—C3	118.2 (3)	C11—C12—H12A	109.5
C1—C2—H2	120.9	C11—C12—H12B	109.5
C3—C2—H2	120.9	H12A—C12—H12B	109.5
C4—C3—C2	120.4 (3)	C11—C12—H12C	109.5
C4—C3—H3	119.8	H12A—C12—H12C	109.5
C2—C3—H3	119.8	H12B—C12—H12C	109.5
C3—C4—C5	121.0 (3)		

O2—S1—N2—C7	−60.0 (3)	C4—C5—C6—C1	−0.5 (4)
O1—S1—N2—C7	170.2 (2)	C2—C1—C6—C5	−1.6 (5)
C4—S1—N2—C7	55.0 (3)	N1—C1—C6—C5	178.0 (3)
C6—C1—C2—C3	2.4 (4)	C8—N3—C7—N4	−1.6 (5)
N1—C1—C2—C3	−177.1 (3)	C8—N3—C7—N2	178.8 (3)

C1—C2—C3—C4	−1.3 (4)	C10—N4—C7—N3	0.8 (4)
C2—C3—C4—C5	−0.8 (5)	C10—N4—C7—N2	−179.6 (3)
C2—C3—C4—S1	176.6 (2)	S1—N2—C7—N3	15.1 (4)
O2—S1—C4—C3	−9.6 (3)	S1—N2—C7—N4	−164.5 (2)
O1—S1—C4—C3	121.6 (2)	C7—N3—C8—C9	1.1 (5)
N2—S1—C4—C3	−127.1 (3)	N3—C8—C9—C10	−0.1 (5)
O2—S1—C4—C5	167.8 (2)	C7—N4—C10—C9	0.5 (4)
O1—S1—C4—C5	−61.1 (3)	C8—C9—C10—N4	−0.8 (5)
N2—S1—C4—C5	50.3 (3)	O3—S2—C11—C12	64.5 (3)
C3—C4—C5—C6	1.7 (5)	O4—S2—C11—C12	−57.7 (3)
S1—C4—C5—C6	−175.7 (2)	O5—S2—C11—C12	−176.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O5^{i}	0.91 (1)	2.05 (1)	2.929 (4)	164 (3)
N1—H2*N*···O4^{ii}	0.90 (1)	2.17 (2)	2.980 (4)	149 (3)
N1—H2*N*···O3^{iii}	0.90 (1)	2.26 (3)	2.705 (3)	110 (2)
N1—H3*N*···O5	0.91 (1)	1.89 (1)	2.801 (3)	176 (3)
N2—H4*N*···N4^{iv}	0.89 (3)	1.99 (3)	2.882 (4)	174 (3)

Symmetry codes: (i) *x*, −*y*+1/2, *z*+1/2; (ii) *x*+1, *y*, *z*; (iii) *x*+1, −*y*+1/2, *z*+1/2; (iv) −*x*+1, −*y*+1, −*z*+1.

(VIII) 4-[(Pyrimidin-2-yl)sulfamoyl]anilinium 4-hydroxybenzenesulfonate dihydrate

Crystal data

$C_{10}H_{11}N_4O_2S^+·C_6H_5O_4S^-·2H_2O$
M_r = 460.48
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 18.5872 (5) Å
b = 5.9733 (2) Å
c = 18.6850 (5) Å
β = 104.858 (3)°
V = 2005.17 (10) Å^3
Z = 4
$F(000)$ = 960
D_x = 1.525 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3724 reflections
θ = 3.3–29.5°
μ = 0.32 mm^{-1}
T = 123 K
Fragment, light yellow
0.20 × 0.12 × 0.05 mm

Data collection

Oxford Diffraction Xcalibur E diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.968, T_{max} = 1.000
9653 measured reflections
4695 independent reflections
3638 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 28.0°, θ_{min} = 3.3°
h = −24→24
k = −7→6
l = −21→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.113
S = 1.03
4695 reflections
307 parameters
6 restraints

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 + 1.8769P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.80$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}

Special details

Experimental. Absorption correction: CrysAlisPro, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.65276 (3)	0.85852 (10)	0.14183 (3)	0.01721 (14)
S2	0.04915 (3)	0.16965 (10)	0.11296 (3)	0.02095 (15)
O1	0.67885 (9)	0.6329 (3)	0.14790 (9)	0.0228 (4)
O2	0.69403 (9)	1.0264 (3)	0.11393 (9)	0.0220 (4)
O1W	0.74103 (10)	1.3663 (3)	0.49790 (10)	0.0294 (4)
O3	0.24507 (11)	0.2558 (3)	0.41461 (10)	0.0307 (4)
O2W	0.38462 (10)	0.1557 (3)	0.45248 (10)	0.0253 (4)
O4	0.09613 (10)	0.1048 (3)	0.06496 (9)	0.0245 (4)
O5	0.01741 (10)	0.3925 (3)	0.09516 (10)	0.0296 (4)
O6	−0.00589 (11)	0.0033 (3)	0.11685 (10)	0.0361 (5)
N1	0.60917 (12)	1.1632 (4)	0.43098 (11)	0.0184 (4)
N2	0.56951 (11)	0.8768 (3)	0.08478 (11)	0.0192 (4)
N3	0.45276 (10)	0.7612 (3)	0.02182 (10)	0.0184 (4)
N4	0.51699 (11)	0.5802 (3)	0.13473 (11)	0.0239 (5)
C1	0.62078 (12)	1.0873 (4)	0.36057 (12)	0.0167 (5)
C2	0.64836 (13)	0.8753 (4)	0.35556 (13)	0.0216 (5)
H2	0.6598	0.7791	0.3974	0.026*
C3	0.65925 (13)	0.8042 (4)	0.28801 (13)	0.0205 (5)
H3	0.6779	0.6584	0.2831	0.025*
C4	0.64263 (12)	0.9487 (4)	0.22827 (12)	0.0153 (4)
C5	0.61737 (13)	1.1644 (4)	0.23418 (13)	0.0201 (5)
H5	0.6084	1.2637	0.1931	0.024*
C6	0.60549 (13)	1.2328 (4)	0.30103 (13)	0.0200 (5)
H6	0.5870	1.3787	0.3060	0.024*
C7	0.51075 (12)	0.7306 (4)	0.08101 (12)	0.0166 (5)
C8	0.39652 (13)	0.6169 (4)	0.01531 (13)	0.0211 (5)

H8	0.3544	0.6301	−0.0259	0.025*
C9	0.39741 (14)	0.4498 (4)	0.06624 (14)	0.0253 (5)
H9	0.3577	0.3457	0.0605	0.030*
C10	0.45878 (14)	0.4411 (4)	0.12597 (14)	0.0270 (6)
H10	0.4599	0.3308	0.1629	0.032*
C11	0.10841 (13)	0.1923 (4)	0.20342 (12)	0.0184 (5)
C12	0.16396 (14)	0.0356 (4)	0.22862 (14)	0.0249 (5)
H12	0.1699	−0.0849	0.1975	0.030*
C13	0.21102 (14)	0.0540 (4)	0.29930 (14)	0.0258 (5)
H13	0.2494	−0.0528	0.3166	0.031*
C14	0.20137 (13)	0.2292 (4)	0.34422 (13)	0.0228 (5)
C15	0.14535 (15)	0.3837 (5)	0.31933 (15)	0.0313 (6)
H15	0.1387	0.5028	0.3507	0.038*
C16	0.09883 (15)	0.3653 (4)	0.24877 (14)	0.0290 (6)
H16	0.0603	0.4719	0.2316	0.035*
H1N	0.6528 (17)	1.235 (5)	0.4588 (16)	0.032 (8)*
H2N	0.5995 (17)	1.043 (6)	0.4561 (17)	0.045 (9)*
H3N	0.5680 (19)	1.260 (5)	0.4221 (17)	0.046 (9)*
H4N	0.5636 (16)	0.987 (5)	0.0532 (16)	0.034 (8)*
H1H	0.292 (2)	0.181 (7)	0.419 (2)	0.088 (15)*
H1W	0.742 (2)	1.482 (4)	0.5266 (16)	0.071 (13)*
H2W	0.7630 (18)	1.420 (5)	0.4655 (14)	0.065 (12)*
H3W	0.4165 (14)	0.081 (4)	0.4344 (17)	0.051 (10)*
H4W	0.4001 (19)	0.295 (2)	0.452 (2)	0.078 (13)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0145 (3)	0.0194 (3)	0.0167 (3)	0.0008 (2)	0.0023 (2)	−0.0025 (2)
S2	0.0188 (3)	0.0259 (3)	0.0179 (3)	−0.0040 (2)	0.0044 (2)	−0.0013 (2)
O1	0.0218 (9)	0.0222 (9)	0.0228 (8)	0.0042 (7)	0.0029 (7)	−0.0054 (7)
O2	0.0187 (8)	0.0270 (9)	0.0205 (8)	−0.0046 (7)	0.0056 (7)	−0.0020 (7)
O1W	0.0253 (10)	0.0363 (11)	0.0280 (10)	−0.0046 (8)	0.0093 (8)	−0.0074 (9)
O3	0.0248 (10)	0.0409 (11)	0.0237 (9)	0.0013 (9)	0.0016 (8)	−0.0093 (8)
O2W	0.0226 (9)	0.0245 (10)	0.0273 (9)	0.0003 (8)	0.0037 (7)	0.0032 (8)
O4	0.0312 (10)	0.0257 (9)	0.0182 (8)	0.0000 (8)	0.0091 (7)	0.0001 (7)
O5	0.0229 (9)	0.0340 (10)	0.0305 (9)	0.0065 (8)	0.0041 (8)	0.0040 (8)
O6	0.0373 (11)	0.0453 (12)	0.0258 (9)	−0.0218 (9)	0.0082 (8)	−0.0038 (9)
N1	0.0186 (10)	0.0190 (10)	0.0185 (10)	−0.0004 (9)	0.0062 (8)	−0.0008 (9)
N2	0.0166 (10)	0.0211 (10)	0.0169 (9)	−0.0022 (8)	−0.0012 (8)	0.0041 (8)
N3	0.0156 (9)	0.0193 (10)	0.0193 (9)	−0.0011 (8)	0.0027 (8)	−0.0014 (8)
N4	0.0217 (11)	0.0232 (10)	0.0255 (11)	−0.0026 (9)	0.0035 (9)	0.0072 (9)
C1	0.0127 (10)	0.0186 (11)	0.0180 (11)	−0.0005 (9)	0.0027 (8)	−0.0024 (9)
C2	0.0248 (12)	0.0208 (12)	0.0185 (11)	0.0048 (10)	0.0041 (9)	0.0051 (10)
C3	0.0238 (12)	0.0149 (11)	0.0224 (12)	0.0048 (9)	0.0050 (10)	0.0000 (9)
C4	0.0121 (10)	0.0172 (11)	0.0159 (10)	0.0004 (9)	0.0021 (8)	−0.0007 (9)
C5	0.0237 (12)	0.0186 (11)	0.0180 (11)	0.0045 (10)	0.0053 (9)	0.0035 (9)
C6	0.0262 (13)	0.0135 (11)	0.0214 (11)	0.0048 (9)	0.0079 (10)	0.0003 (9)

C7	0.0161 (11)	0.0159 (11)	0.0179 (11)	0.0010 (9)	0.0043 (9)	−0.0017 (9)
C8	0.0175 (11)	0.0232 (12)	0.0218 (12)	−0.0007 (10)	0.0038 (9)	−0.0037 (10)
C9	0.0202 (12)	0.0225 (12)	0.0339 (14)	−0.0061 (10)	0.0079 (10)	0.0011 (11)
C10	0.0249 (13)	0.0244 (13)	0.0314 (13)	−0.0019 (11)	0.0070 (11)	0.0087 (11)
C11	0.0169 (11)	0.0211 (12)	0.0174 (11)	−0.0024 (9)	0.0050 (9)	−0.0028 (9)
C12	0.0254 (13)	0.0248 (13)	0.0252 (12)	0.0035 (11)	0.0075 (10)	−0.0079 (10)
C13	0.0207 (12)	0.0280 (13)	0.0277 (13)	0.0066 (11)	0.0042 (10)	0.0002 (11)
C14	0.0198 (12)	0.0286 (13)	0.0194 (11)	−0.0058 (10)	0.0042 (9)	−0.0064 (10)
C15	0.0320 (15)	0.0299 (14)	0.0301 (14)	0.0051 (12)	0.0046 (11)	−0.0141 (11)
C16	0.0292 (14)	0.0272 (14)	0.0280 (13)	0.0079 (11)	0.0026 (11)	−0.0056 (11)

Geometric parameters (Å, º)

S1—O1	1.4271 (17)	C1—C6	1.383 (3)
S1—O2	1.4390 (17)	C2—C3	1.395 (3)
S1—N2	1.6422 (19)	C2—H2	0.9500
S1—C4	1.758 (2)	C3—C4	1.382 (3)
S2—O6	1.4413 (19)	C3—H3	0.9500
S2—O4	1.4557 (18)	C4—C5	1.386 (3)
S2—O5	1.4597 (19)	C5—C6	1.385 (3)
S2—C11	1.770 (2)	C5—H5	0.9500
O1W—H1W	0.873 (10)	C6—H6	0.9500
O1W—H2W	0.874 (10)	C8—C9	1.376 (3)
O3—C14	1.366 (3)	C8—H8	0.9500
O3—H1H	0.96 (4)	C9—C10	1.377 (3)
O2W—H3W	0.875 (10)	C9—H9	0.9500
O2W—H4W	0.880 (10)	C10—H10	0.9500
N1—C1	1.459 (3)	C11—C16	1.376 (3)
N1—H1N	0.95 (3)	C11—C12	1.384 (3)
N1—H2N	0.90 (3)	C12—C13	1.389 (3)
N1—H3N	0.94 (3)	C12—H12	0.9500
N2—C7	1.386 (3)	C13—C14	1.382 (4)
N2—H4N	0.87 (3)	C13—H13	0.9500
N3—C8	1.336 (3)	C14—C15	1.380 (4)
N3—C7	1.344 (3)	C15—C16	1.383 (3)
N4—C7	1.330 (3)	C15—H15	0.9500
N4—C10	1.340 (3)	C16—H16	0.9500
C1—C2	1.378 (3)		
O1—S1—O2	119.01 (10)	C5—C4—S1	118.46 (17)
O1—S1—N2	111.03 (10)	C6—C5—C4	118.8 (2)
O2—S1—N2	102.68 (10)	C6—C5—H5	120.6
O1—S1—C4	109.32 (10)	C4—C5—H5	120.6
O2—S1—C4	108.18 (10)	C1—C6—C5	119.5 (2)
N2—S1—C4	105.72 (10)	C1—C6—H6	120.2
O6—S2—O4	113.15 (11)	C5—C6—H6	120.2
O6—S2—O5	112.94 (12)	N4—C7—N3	127.5 (2)
O4—S2—O5	111.94 (11)	N4—C7—N2	118.2 (2)

O6—S2—C11	106.29 (11)	N3—C7—N2	114.3 (2)
O4—S2—C11	106.50 (11)	N3—C8—C9	122.3 (2)
O5—S2—C11	105.31 (11)	N3—C8—H8	118.8
H1W—O1W—H2W	101 (2)	C9—C8—H8	118.8
C14—O3—H1H	109 (3)	C8—C9—C10	116.6 (2)
H3W—O2W—H4W	103 (2)	C8—C9—H9	121.7
C1—N1—H1N	109.4 (17)	C10—C9—H9	121.7
C1—N1—H2N	109 (2)	N4—C10—C9	123.4 (2)
H1N—N1—H2N	109 (3)	N4—C10—H10	118.3
C1—N1—H3N	109.5 (19)	C9—C10—H10	118.3
H1N—N1—H3N	111 (3)	C16—C11—C12	120.1 (2)
H2N—N1—H3N	109 (3)	C16—C11—S2	119.87 (19)
C7—N2—S1	126.17 (17)	C12—C11—S2	120.04 (18)
C7—N2—H4N	118.7 (19)	C11—C12—C13	120.2 (2)
S1—N2—H4N	115 (2)	C11—C12—H12	119.9
C8—N3—C7	115.5 (2)	C13—C12—H12	119.9
C7—N4—C10	114.5 (2)	C14—C13—C12	119.3 (2)
C2—C1—C6	121.7 (2)	C14—C13—H13	120.4
C2—C1—N1	119.4 (2)	C12—C13—H13	120.4
C6—C1—N1	118.9 (2)	O3—C14—C15	117.2 (2)
C1—C2—C3	118.9 (2)	O3—C14—C13	122.3 (2)
C1—C2—H2	120.5	C15—C14—C13	120.4 (2)
C3—C2—H2	120.5	C14—C15—C16	120.1 (2)
C4—C3—C2	119.1 (2)	C14—C15—H15	120.0
C4—C3—H3	120.4	C16—C15—H15	120.0
C2—C3—H3	120.4	C11—C16—C15	119.9 (2)
C3—C4—C5	121.8 (2)	C11—C16—H16	120.1
C3—C4—S1	119.76 (17)	C15—C16—H16	120.1

O1—S1—N2—C7	42.5 (2)	S1—N2—C7—N4	10.8 (3)
O2—S1—N2—C7	170.77 (19)	S1—N2—C7—N3	−169.56 (17)
C4—S1—N2—C7	−75.9 (2)	C7—N3—C8—C9	0.8 (3)
C6—C1—C2—C3	−1.7 (4)	N3—C8—C9—C10	1.6 (4)
N1—C1—C2—C3	180.0 (2)	C7—N4—C10—C9	0.6 (4)
C1—C2—C3—C4	0.5 (4)	C8—C9—C10—N4	−2.3 (4)
C2—C3—C4—C5	1.8 (4)	O6—S2—C11—C16	−99.8 (2)
C2—C3—C4—S1	−177.68 (18)	O4—S2—C11—C16	139.3 (2)
O1—S1—C4—C3	−0.2 (2)	O5—S2—C11—C16	20.3 (2)
O2—S1—C4—C3	−131.21 (19)	O6—S2—C11—C12	79.8 (2)
N2—S1—C4—C3	119.37 (19)	O4—S2—C11—C12	−41.1 (2)
O1—S1—C4—C5	−179.72 (17)	O5—S2—C11—C12	−160.1 (2)
O2—S1—C4—C5	49.3 (2)	C16—C11—C12—C13	−1.0 (4)
N2—S1—C4—C5	−60.1 (2)	S2—C11—C12—C13	179.4 (2)
C3—C4—C5—C6	−2.9 (4)	C11—C12—C13—C14	0.5 (4)
S1—C4—C5—C6	176.64 (18)	C12—C13—C14—O3	179.2 (2)
C2—C1—C6—C5	0.6 (4)	C12—C13—C14—C15	0.3 (4)
N1—C1—C6—C5	179.0 (2)	O3—C14—C15—C16	−179.6 (3)
C4—C5—C6—C1	1.6 (4)	C13—C14—C15—C16	−0.7 (4)

C10—N4—C7—N3	2.2 (4)	C12—C11—C16—C15	0.7 (4)
C10—N4—C7—N2	−178.2 (2)	S2—C11—C16—C15	−179.7 (2)
C8—N3—C7—N4	−2.9 (4)	C14—C15—C16—C11	0.1 (4)
C8—N3—C7—N2	177.5 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1*W*	0.95 (3)	1.80 (3)	2.733 (3)	170 (3)
N1—H2*N*···O2*W*^{i}	0.90 (3)	2.04 (3)	2.873 (3)	154 (3)
N1—H2*N*···O5^{ii}	0.90 (3)	2.32 (3)	2.793 (3)	113 (2)
N1—H3*N*···O6^{iii}	0.94 (3)	1.88 (3)	2.780 (3)	159 (3)
N2—H4*N*···N3^{iv}	0.87 (3)	2.03 (3)	2.897 (3)	179 (3)
O3—H1*H*···O2*W*	0.96 (4)	1.69 (4)	2.578 (3)	153 (4)
O1*W*—H1*W*···O3^{v}	0.87 (1)	1.89 (1)	2.760 (3)	173 (3)
O1*W*—H2*W*···O2^{vi}	0.87 (1)	1.96 (1)	2.834 (3)	175 (3)
O2*W*—H3*W*···O5^{vii}	0.88 (1)	1.85 (1)	2.722 (3)	172 (3)
O2*W*—H4*W*···O4^{ii}	0.88 (1)	1.88 (1)	2.737 (2)	163 (4)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1/2, *y*+1/2, −*z*+1/2; (iii) −*x*+1/2, *y*+3/2, −*z*+1/2; (iv) −*x*+1, −*y*+2, −*z*; (v) −*x*+1, −*y*+2, −*z*+1; (vi) −*x*+3/2, *y*+1/2, −*z*+1/2; (vii) −*x*+1/2, *y*−1/2, −*z*+1/2.